BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 8, 1926.

I.—GENERAL; PLANT; MACHINERY.

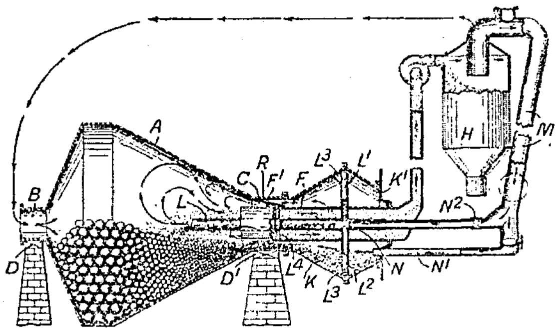
New methods of gas washing. IV. Absorption curves with mixed absorbents. G. Weissenberger, F. Schuster, and O. Zack (Z. angew. Chem., 1925, 38, 1010—1013; *cf.* B., 1925, 388, 697).—In certain cases of the extraction of vapour from a gas by washing with a liquid which forms with the vapour a molecular compound, it is necessary to add a second liquid to the " extractor " which, while taking no part in the primary absorption, protects the extractor against some harmful secondary reaction. Thus in the extraction of methyl acetate by phenol the dilution of the latter with tetralin protects it against rapid resinification. The vapour pressures at different concentrations of the following systems were determined:—vapours: methyl acetate and ethyl acetate; extractor; phenol; protectors: tetralin and *cyclo*hexanol; vapour: chloroform; extractor: tetralin; protector: phenol. The choice of the " protector " modifies greatly the experimentally determined vapour pressures. For the absorption of methyl acetate in phenol much better results will be obtained with the use of tetralin than of *cyclo*hexanol. Quite different results are found in the case of ethyl acetate vapour. A further variation occurs in the absorption of chloroform in tetralin with phenol as protector. In this case the protector has only a very slight influence on the process. C. Irwin.

[Use of] ethyleneglycol [as anti-freeze material]. G. O. Curme, jun., and C. O. Young (Ind. Eng. Chem., 1925, 17, 1117—1120).—Ethyleneglycol, which is now being produced in large quantities, is satisfactory for use as anti-freeze material in motor car radiators. It is non-corrosive, odourless, and non-inflammable; it does not vaporise or decompose, nor does it attack lacquer or varnish. A 35% solution in water freezes at about $-20°$. L. A. Coles.

Patents.

Refrigerating apparatus of the absorption type. Platen-Munters Refrigerating System Aktiebolag (E.P. 231,521, 28.3.25. Conv., 29.3.24).—The patent describes various forms of cooler foi an absorption refrigerating system in which an inert carrier gas is used as well as the vaporising liquid. The coolers combine large surface for evaporation, good heat conductivity to the outside air, and absence of obstruction to the flow of gas. B. M. Venables.

Dry grinding or crushing apparatus. H. W. Hardinge (E.P. 241,268, 15.7.24).*—A cylindro-conical ball or pebble mill is provided with a device for the separation of dust and return of oversize as shown in the figure. F is an eduction conduit for air and finely-ground material, the part of which to



the left of the stationary disc, K^1, rotates with the mill, whilst the part to the right is fixed. The conical classifier, K, with the pipes, L^1, L^2, and L, also rotate with the mill and together serve to return partially ground material to a point in the mill, A, where it may most effectively be re-ground, the pipes L^1, L^2 being bent to form scoops. The dusty air is drawn by a fan and delivered to a collector, H, and practically clean air is returned to the mill through the conduit, M, which branches into two conduits, N^2 and N^1. The air stream in N^2 passes through a nozzle and assists the oversize on its way back to the mill. The air in N^1 is blown over the surface of the material in the classifier, K, and returns any dust to the mill at a point where it can easily be collected by the stream of air going out through F. The trunnion outlet, C, is provided with inclined shallow helical ribs, R, to return any balls, pebbles, or chips to the mill, without interfering with the output of partially-ground material. B. M. Venables.

Viscosimeters. G. G. STONEY and R. O. BOSWALL (E.P. 241,652, 12.8.24).—A viscosimeter suitable for measuring the viscosity of oil in the feed of an oil engine, or for other similar purpose, comprises a tube and an orifice in series, with means for measuring the fall of pressure between the inflow and exit ends of the tube and orifice. If the flow is so adjusted that the pressure fall at the orifice is constant, then the fall between the inflow and exit ends of the tube bears a simple relation to the viscosity and affords a measure of the latter. A. B. MANNING.

Evaporators. H. FOTHERGILL (E.P. 241,671, 4.9.24).—In a flash evaporator in which the liquid to be evaporated is heated at a high pressure and evaporated at a lower pressure, the difference in pressure is produced by the friction of the liquid in the conduits and/or the spraying jets. B. M. VENABLES.

Evaporation of solutions of salt or other substances to produce crystals. R. FRANCE (E.P. 242,018, 23.8 and 19.12.24).—The solution is circulated through a heater and evaporating pan by means of a pump or by thermo-siphon action. The pan, which is open to the atmosphere, is not heated but is reserved for evaporation and removal of crystals, and baffles are provided to prevent deposited crystals entering the return pipe. The heater may take the form of a cylindrical vessel with a nest of heating tubes situated below the evaporating pan, and with a cylindrical extension passing up through the bottom of the pan with perforations in its upper edge through which the heated liquor can emerge on to the surface of the liquor in the pan. B. M. VENABLES.

Furnaces for burning pulverised fuel. UNDERFEED STOKER CO., and W. R. WOOD (E.P. 241,691, 26.9.24).—Spaced cooling tubes are arranged at the bottom of the furnace, and are connected to a "header" from the boiler. The tubes are built into the form of a cone and the fine ash particles collect on this, become chilled, and fall to an ash hopper at the bottom of the combustion chamber. The lower part of the walls of the combustion chamber are also cooled by the insertion of "fin tubes" which prevent any ash fusing to the walls. The extra heating surface provided by the tubes allows of a material reduction of the boiler proper. W. S. PATTERSON.

Method of conveying heat energy. J. N. D. HEENAN, Assr. to POWER SPECIALTY CO. (U.S.P. 1,558,871, 27.10.25. Appl., 11.12.18).—A fluid is heated indirectly from a source of heat by means of a fluid heat-transferring medium which flows in a conduit arranged as pipe coils alternately in a flue connected with the source of heat and in the material to be heated. The coils in the material to be heated are so arranged that each coil has a greater exposed area than the one preceding it, in the direction of flow of the heat-transferring medium, and the coils which are being heated are so arranged that each has a greater area than any other nearer the source of heat. B. M. VENABLES.

Heating method and apparatus. F. PUENING (U.S.P. 1,542,953–7, 23.6.25. Appl., 16.7.20, 11.12.22, 18.1.23, 31.1.23, and 23.1.24).—See E.P. 208,170, 210,068, and 210,758; B., 1925, 527, 867. The additional details given relate mainly to means of keeping the piston cool. B. M. VENABLES.

Apparatus for extraction, solution, and mixture of soluble and insoluble substances. N. BENDIXEN, W. E. MCKECHNIE, and E. L. REID (E.P. 242,020, 27.8 and 11.11.24).—A propeller rotates in a chamber bounded by a vertical serrated or corrugated and perforated wall, the serrations being parallel to the axis. Outside the propeller chamber is an intermediate wall and outside of all, the non-perforated casing or liquid container. When solid substances are to be extracted by the flow of liquid through them, the intermediate wall is perforated and situated some distance from the inner wall, the solid matter being contained in the annular space between the two, but when emulsions are to be formed from substances that are already liquid or finely divided, the intermediate wall need not be perforated but should be arranged to touch the ridges of the serrations of the inner wall, thus forming a number of vertical passages. The propeller drives the liquid both outwards and downwards and a vertical flow is produced in the passages between the two walls which has the effect of shearing the particles or globules that are driven outwards from the propeller chamber. B. M. VENABLES.

Drying apparatus. D. V. SHERBAN, Assr. to BONNOT CO. (U.S.P. 1,558,119, 20.10.25. Appl., 6.11.24).—The material to be dried is delivered into a hopper provided at its lower end with spaced ring plates. A series of conical plates, spaced from the ring plates, is rotated, and means are provided for passing hot gases between the conical plates and the ring plates. H. HOLMES.

Treatment of liquids with gases. CHEM. FABR. NIEDERRHEIN GES., and K. ULLMANN (G.P. 417,507, 13.11.23).—The gas, under pressure less than that of the hydrostatic pressure of the liquid, is introduced into the liquid, and suction applied by means of a rotary pump, so that the liquid is drawn through the pump after the gas. The mixture is then forced through a long channel in which the rate of flow of the gas differs from that of the liquid. B. W. CLARKE.

Air-cleaning [filter] materials. REED AIR FILTER CO., Assees. of C. P. HEGAN (E.P. 231,435, 6.2.25. Conv., 27.3.24).—See U.S.P. 1,515,949; B., 1925, 32.

Operation of reverberatory furnaces. Flame furnace.—H. MEHNER (U.S.P. 1,545,154–5, 7.7.25. Appl., 14.2.22 and 10.4.25).—See E.P. 196,658; J., 1923, 589A.

Thickening [filtering] process. A. L. GENTER, Assr. to GEN. ENGINEERING CO. (U.S.P. 1,560,796, 10.11.25. Appl., 2.1.24).—See E.P. 227,094; B., 1925, 698.

Grinding mill. J. B. Sedberry (U.S.P. 1,560,865, 10.11.25. Appl., 28.11.23. Renewed 10.4.25).—See E.P. 229,034 ; B., 1925, 304.

Coal dust furnaces. G. Hayn (E.P. 241,882, 8.10.25. Conv., 24.10.24).

Purifier for [separating suspended particles from] air or gas. C. L. Burdick (E.P. 242,388, 12.9.24).

Mixing and kneading machines. Haagen & Rinau, and W. Rinau (E.P. 242,573, 28.8.25. Conv., 21.2.25).

Crushing discs. E. Symons (E.P. 242,831, 2.2.25).

Separation of liquids from gases. H. S. Hele-Shaw and T. E. Beacham (E.P. 242,918, 29.12.24).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Examination of coal and grain size of samples for analysis. D. J. W. Kreulen (Chem. Weekblad, 1925, **22**, 558—560 ; *cf.* B., 1924, 775, 896).—Various tests applied after different mixing and sieving operations show that results vary with the grain size and degree of mixing. Material passed through a 10-mesh sieve gave results varying considerably, according to the procedure adopted in mixing after sieving, but material passed through sieves of finer mesh gives uniform results. S. I. Levy.

Mixing of coal samples and methods of obtaining final samples. D. J. W. Kreulen (Chem. Weekblad, 1925, **22**, 560—561 ; *cf.* B., 1924, 83, and preceding abstract).—Various methods of mixing and of drawing small samples for analysis from the mixed material have been examined by comparison of analytical results in the various cases. The American method of spreading out in a long layer and taking scoopfuls alternately from left to right and from right to left gives the best results. S. I. Levy.

Oily bitumen and solid bitumen of bituminous coal. H. Broche and T. Bahr (Brennstoff-Chem., 1925, **6**, 349—354 ; *cf.* B., 1924, 584.). Bone (J., 1925, 291 T) has suggested that the apparent discrepancy between his results and those of Fischer, Broche, and Strauch is due to the incomplete separation of the constituents of the benzene extract by the latter. A repetition and extension of the work shows this not to be the case, and confirms the conclusion that the oily bitumen is mainly responsible for the coking (*i.e.*, binding) property of the original coal, but that the swelling is due to the solid bitumen. Bone's results on the addition of the bitumen to powdered coke have been confirmed, and the discrepancy shown to lie in the use of powdered coke instead of the residue after extraction when investigating the relation of the bitumen constituents to coking property. On addition of the extracted bitumen to the residue the full coking properties of the original coal are recovered, but are almost completely lost if the residue is replaced by powdered coke. Misleading results are obtained also if the particle size of the residue and conditions of coking are not carefully controlled. If the residue is powdered too finely before or after addition of the bitumen, the coking power is not fully recovered. Some further experiments have been made on the separation and purification of the bitumen constituents. Extraction of the coal with light petroleum (b.p. 65—90° or 100—125°) under pressure at 285° removes very little of the coking constituents. A. B. Manning.

[Steel] bomb calorimeters. W. Kohen (Chem.-Ztg., 1925, **49**, 935—936).—Plain steel bomb calorimeters with enamel linings have the disadvantage that tho lining wears out comparatively rapidly. Calorimeters made of V2A steel, which contains nickel resist the action of the products of combustion quite well, but have a low thermal conductivity. Rustless (stainless) steel is even more resistant to corrosion than V2A steel, and has a much better heat conductivity. During the first few combustions a very slight amount of iron (about 1—2 mg.) is dissolved, but with use an inert film is formed on the surface and no further attack takes place unless the substance burnt contains chlorine or much sulphur. The best steel for the making of bomb calorimeters contains 0·14% C, 0·496% Si, 16·5% Cr, and a trace of manganese ; it is used in Germany under the trade name of " Antinit." A. R. Powell.

Determination of hydrogen and methane in illuminating gas. W. Steuer (Chem.-Ztg., 1925, **49**, 901—902).—After absorption of carbon monoxide in the usual way with ammoniacal cuprous chloride solution, the Hempel or Winkler burette containing the remaining gas is connected to a second burette, containing a known volume of pure oxygen, by means of a capillary quartz tube inside which is a thick platinum or palladium wire. The quartz tube is heated very gently with a small flame and the mixed gases are passed backwards and forwards until constant volume is attained. The metal acts as a catalyst ; the hydrogen only is burned and is determined by the contraction measured. The capillary is then heated to a clear red heat, and the process repeated. The amount of methane can be calculated from the contraction in volume or the carbon dioxide formed can be absorbed. Alternatively, as soon as most of the hydrogen has combined, the capillary is strongly heated and the remaining hydrogen and the methane are burned at the same time, without risk of explosion. The method is rapid and accurate. C. T. Gimingham.

Corrosion of copper pipe by kerosene. E. Staudt (Chem.-Ztg., 1925, **49**, 952).—A spiral copper pipe leading kerosene to the carburettor and heated externally by the exhaust gases, was installed on a tractor. After two weeks the pipe was found to be almost filled with a grey-black mass, and the walls of the pipe were reduced to 0·1 mm. in thickness. The deposit contained 1% of copper sulphate, and

the remainder corresponded to the formula Cu_2S. The kerosene had a sulphur content of 0·104%, and as 40 litres passed through the pipe daily this was quantitatively sufficient for the formation of the deposit. The deposit, being porous, did not form a protective layer. Copper is unsafe for conduits for petroleum oils at elevated temperatures.

H. Moore.

Gas washing. Absorption with mixed absorbents. Weissenberger, Schuster, and Zack.—See I.

Mechanical condition of ammonium sulphate. Cummings.—See VII.

Patents.

Briquetting fuels. Frankensteiner Magnesitwerke A.-G., Assees. of R. Kattner (E.P. 241,175, 14.9.25. Conv., 11.10.24. Not yet accepted).—In utilising magnesia cement for briquetting fuel, the coal dust or the like is first treated with ground magnesium chloride (1%), and an amount of water necessary for briquetting is added. Calcined magnesia (2%) is only added when the mixture is ready to be moulded.

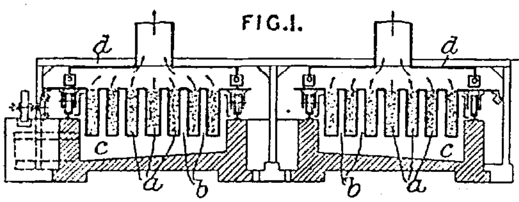
Production of fuel of high calorific value from semi-coke. E. C. R. Marks. From Kohlenscheidungs-Ges.m.b.H. (E.P. 241,262, 11.7.24).—Coke obtained by the low-temperatire distillation of fuel is pulverised and treated for the removal of ash. The product may be briquetted or suspended in oil with the addition of a protective colloid.

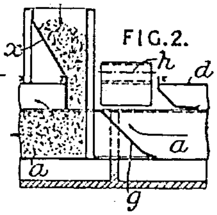
H. Hollings.

Coke ovens. Soc. Gén. de Fours à Coke Systèmes Lecocq (E.P. 231,483, 23.3.25. Conv., 29.3.24).—Even heating in coke ovens of great height is obtained by admitting the necessary air for combustion into the vertical heating flues at two stages. The primary air and the secondary air are admitted independently to the conduits in the heating walls from two hearth flues extending the whole length of the oven. The passage of air from the regenerators to each hearth flue is controlled by dampers. The burnt gases are exhausted partly through conduits in the heating walls opening into a third flue in the hearth and partly through the base of the flues in the heating walls into a fourth flue in the hearth.

A. B. Manning.

Annular kilns [for drying and carbonising carbonaceous materials]. O. Dobbelstein and H. Hess (E.P. 228,532, 23.1.25. Conv., 2.2.24).*—An annular kiln comprises a rotary system of concentric ring pockets, *a*, separated by ring spaces, *b*, communicating with a stationary annular heating channel, *c*, situated below the pockets. The material is fed into the pockets from a hopper, *x*, and after treatment, is scraped out by a bucket elevator or by ploughs, *g*, which deliver it to a radially arranged conveyor, *h*. The casing, *d*, may be divided by radial partitions so that the distillation products can be collected separately at different stages. In a modification the system of pockets is stationary and the casing, *d*, with the feed hopper and discharge ploughs

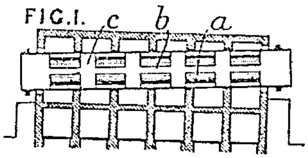




rotates. In another form the bottoms of the pockets, *a*, are formed by stationary plates having gaps at the discharging point.

B. W. Clarke.

[Destructive] distillation drums. Schwelkohle Kohlenschwelungsges., and D. Young (E.P. 240,795, 7.7.25. Conv., 3.10.24. Not yet accepted).*—



In an inclined distillation drum the inner surface is provided with chambers, *a*, formed by partitions running longitudinally and transversely, these chambers carrying the charge round in them during the rotation of the drum. The chambers may either have circumferential spaces, *b*, between them or may be made in a closed ring. Longitudinally, the chambers are spaced apart at intervals, *c*.

Distillation or heat treatment of carbonaceous and other materials. H. Nielsen and B. Laing (E.P. 241,659, 20.8.24).—A small retort for the laboratory assay of carbonaceous material comprises an outer and an inner vertical cylindrical chamber, surrounded by a coil through which a heating medium (steam, combustion gases, or a heated inert gas) can be conducted, the medium then passing down the annular space between the two chambers, and up through the perforated base-plate of the inner chamber. In the latter, it comes into direct contact with the material to be carbonised, and thence carries away the volatile products of decomposition to a condensing system. Auxiliary heating, when required, is provided by a gas burner at the base of the outer chamber. A slight modification of the apparatus makes it suitable for the treatment of liquid materials.

A. B. Manning.

Apparatus for the simultaneous production of low-temperature tar, semi-coke, and gas of high calorific value. N. Young (G.P. 414,185, 18.2.20. Addn. to 408,111).—Coal is passed through a rotating dryer before it enters the distillation retort in the apparatus described in the chief patent (*cf.* E.P. 134,529; J., 1921, 74 A). The dryer and retort are co-axial, are heated independently, and are connected with suction apparatus so operated that a portion of the steam generated in the dryer passes through the retort. The addition of the dryer increases the output of the retort.

L. A. Coles.

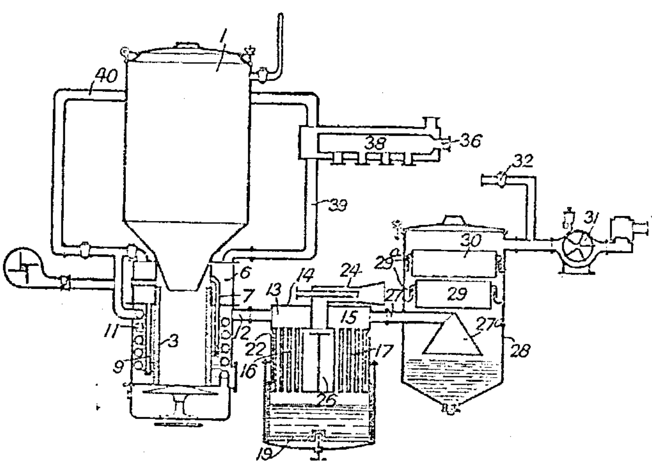
Manufacture of prepared charcoal. A. W. Smith, Assr. to W. W. Varney (U.S.P. 1,559,054, 27.10.25. Appl., 10.3.20).—A charcoal for decolorising, absorption, etc., is prepared by carbonising vegetable material which has been impregnated with a metal compound, the latter then being reduced to the metallic state. A. B. Manning.

Destructive distillation of wood. D. P. Shaw (U.S.P. 1,559,994, 3.11.25. Appl., 21.12.22).—The wood is progressively burnt from one end to the other of a kiln in a horizontal direction, and the distilled vapours are withdrawn from a zone in advance of the progressively burning portion, so as substantially to avoid intermixture with the gases of combustion. A. B. Manning.

Apparatus for wood distillation. J. L. Weaver (U.S.P. 1,560,517, 3.11.25. Appl., 21.3.21).—A retort for the distillation of wood is provided with a helical flue and a stack communicating with the flue in different horizontal planes, communication being controlled by means of dampers. A. B. Manning.

Desulphurising spent absorption charcoal. Badische Anilin- u. Soda-Fabr. (G.P. 417,614, 15.7.24).—The charcoal is heated to different temperatures in separate retorts, in such a manner that sulphur liberated by heating one portion of the charcoal reacts with another portion of the charcoal with the formation of carbon disulphide. L. A. Coles.

Semi-water-gas producer plants. R. Malbay (E.P. 224,520, 20.10.24. Conv., 9.11.23).*—Gas made in a generator, 3, passes down circularly disposed tubes, 7, into the space surrounding the coil, 11, and thence through the outlet, 12, down the tubes, 16, and up the tubes, 17, of a cooler, air being drawn



over the tubes and through a chamber, 26, by means of an injector, 24, operated by the exhaust of an engine. The gas is then directed on to the surface of water by means of the tapered pipe, 27*a*, and passes through suspended coke filters, 29 and 30. The gas, thus purified, is mixed with air drawn in through the valve, 32, compressed in the compressor, 31, and supplied to the engine. The air supplied to the generator is drawn in through the opening, 36, preheated by contact with the engine exhaust collector, 38, and passed by a pipe, 39, to a boiler, 6, the air and steam from which meet an additional air supply from a pipe, 40, and are superheated in the coil, 11, before entering the bottom of the fuel bed. Field tubes, 9, depending from the boiler assist in heating the water. H. Hollings.

Manufacture of illuminating gas. L. S. Stiles (U.S.P. 1,558,124, 20.10.25. Appl., 10.6.20).—A rich carburetted water-gas is made in a separate generator fitted with a steam supply at the bottom and an oil supply at the top and is delivered into the carburettor of a plant making blue water-gas. H. Hollings.

Water-gas apparatus. L. S. Stiles (U.S.P. 1,558,125, 20.10.25. Appl., 18.8.21).—A vessel comprising an upper superheater chamber and a lower carburettor chamber is superimposed upon a gas generator. Oil is supplied to a chamber between the upper and lower portions of chequer brickwork placed between the carburettor and the superheater. Air for combustion is admitted to the carburettor and superheater during the heating-up process. The superheater is provided with a valve-controlled outlet for waste gas and a separate outlet in communication with a wash-box for water-gas. H. Hollings.

Gas purification and regenerating sulphided alkaline solutions. Koppers Co., Assees. of R. A. Morgen, W. S. Yard, and L. Rosenstein (E.P. 241,221, 10.10.25. Conv., 11.10.24. Not yet accepted). —Liquors containing sulphides, for example those produced in a gas-purifying process, are oxidised in the presence of a catalyst (nickel sulphide, cobalt sulphide) whereby the liquors are freed from sulphur, which separates in the elementary state.

Elimination of hydrogen sulphide from fuel gases such as coal gas, water-gas, and the like, and from air. Treatment of spent liquids of liquid gas purification. P. C. Rushen. From Koppers Co. (E.P. (a) 241,248 and (b) 241,452, 10.6.24.)—(a) Air, which may result from the purification of gas as described in U.S.P. 1,389,980 and 1,390,037 (J., 1921, 762 a), or gas is washed with a suspension of a freshly precipitated iron compound, such as the hydroxide, carbonate, or sulphide, in an alkaline solution. The washing of air may take place in a stack fitted with spaced hurdles. The iron compound may be precipitated by the addition from time to time of an iron salt to the circulating liquid or it may be prepared externally. The alkalinity of the liquid is maintained equivalent to 0·5—2·0% Na_2CO_3. (b) The spent wash liquid is collected in a sump fitted with a horizontal partition of porous material such as "filtros" through which compressed air is passed upwards for the regeneration of the suspension. The sulphur carried to the surface of the liquid by the air is removed by skimming. The sump may be

divided into compartments to each of which air is supplied from a manifold. H. Hollings.

Scrubbing hot gases for the recovery of hydrocarbons. Allgem. Vergasungs-Ges.m.b.H. (G.P. 416,815, 21.12.17.)—Hydrocarbons are extracted from gases at a temperature of, *e.g.*, 200—400°, by treating the gases, without previous cooling, with finely-divided tar and a cold, readily volatile liquid, such as water, in a rotary scrubbing apparatus. The resulting tar has a low water content. L. A. Coles.

Deodorising kerosene. W. Kritshevsky and H. C. Prutzman, Assrs. to W. Citron (U.S.P. 1,538,287, 19.5.25. Appl., 2.7.24).—Kerosene is washed with chlorosulphonic acid, then with alkali, and finally treated with a solid absorbent colloid. T. A. Smith.

Dehydrating oil. H. F. Owen (U.S.P. 1,538,739, 19.5.15. Appl., 10.12.23).—Oil is passed through a heater in a continuous stream, being whirled during the heating process. T. A. Smith.

Medium for bleaching, cracking and desulphurising petroleum and other hydrocarbon compounds. H. and H. Reinbold (U.S.P. 1,558,631–2, 27.10.25. Appl., 19.5.24).—(a) A medium for treating oils comprises hydrous silicic acid in combination with lithium chloride. (b) A medium comprising lithium chloride combined with aluminium chloride for treating oils is prepared by treating an alumino-silicate clay with hydrochloric acid and adding lithium chloride. H. Moore.

Condensing [hydrocarbon] vapours. E. W. Isom and J. E. Bell, Assrs. to Sinclair Refining Co. (U.S.P. 1,558,811, 27.10.25. Appl., 12.11.24).—The vapours are condensed by a cooled body of condensate of corresponding composition. A stream of condensate is circulated to and from the main body through an external circuit, the circulation being maintained by the injection of the vapours into the stream. H. Moore.

[Liquid] fuel. J. F. P. de la Riboisiere (U.S.P. 1,558,967, 27.10.25. Appl., 17.12.24).—The heat units obtainable from a heavy hydrocarbon fuel are increased by burning the fuel in the presence of a small proportion of a compound of a hydrocarbon radical with a metal. M. Cook.

Dehydrating emulsified oils. G. Egloff and H. P. Benner, Assrs. to Universal Oil Products Co. (U.S.P. 1,559,035, 27.10.25. Appl., 1.11.20. Renewed 28.3.25).—Heated emulsified petroleum oil is passed, under pressure, through a series of plates having perforations of decreasing size, and is simultaneously subjected to the action of an electric current. H. Moore.

Dehydration of oil. G. Egloff and J. C. Morrell, Assrs. to Universal Oil Products Co. (U.S.P. 1,559,036, 27.10.25. Appl., 18.2.22. Renewed 28.3.25).—A medium adapted to improve the conductivity is dissolved in the oil and water, and an electric current passed through. The water is then separated from the oil in a settling tank. H. Moore.

Manufacture of fuel briquettes. T. A. Goskar (U.S.P. 1,561,322, 10.11.25. Appl., 26.6.25).—See E.P. 231,934; B., 1925, 435.

Annular kiln for drying and carbonising solid carbonaceous or like material. O. Dobbelstein and H. Hess (U.S.P. 1,556,571, 6.10.25. Appl., 24.1.25).—See E.P. 228,532; preceding.

Retort for carbonisation of solid fuels. A. L. J. Queneau, Assr. to E. Heisch (U.S.P. 1,560,855, 10.11.25. Appl., 27.11.23).—See E.P. 221,679; B., 1924, 974.

Retort furnace. O. D. Lucas, Assr. to Vickers, Ltd. (U.S.P. 1,561,735, 17.11.25. Appl., 31.3.24).—See E.P. 220,684; B., 1924, 899.

Tar separator. B. Moscovitch, Assr. to "Lignojen" Maschinen- u. Apparatebau Ges. (U.S.P. 1,562,118, 17.11.25. Appl., 18.10.24).—See E.P. 239,065; B., 1925, 872.

Filtering liquids [mineral oils etc., with the aid of silicic acid]. F. Herrmann (E.P. 216,504, 5.5.24. Conv., 23.5.23).—See G.P. 402,508; B., 1925, 58.

Apparatus for carburetting coal gas [with petrol or the like]. E. McLaren (E.P. 242,553, 16.6.25).

Viscosimeters. (E.P. 241,652).—See I.

Bituminous binding materials. (E.P. 233,371).—See IX.

Bituminous emulsions. (E.P. 233,430).—See IX.

Vulcanised rubber. (G.P. 416,877).—See XIV.

III.—TAR AND TAR PRODUCTS.

Vulcanised rubber. (G.P. 416,877).—See XIV.

Disinfectants. (G.P. 416,599).—See XXIII.

IV.—DYESTUFFS AND INTERMEDIATES.

Composition of some products used for the production of insoluble azo colours. F. M. Rowe and E. Levin (J. Soc. Dyers and Col., 1925, **41**, 354—356; *cf.* B., 1925, 397).—Naphthol AS-BR (GrE) which is used with various diazotised amines for the production of rich brown shades, is the dianisidide of β-hydroxynaphthoic acid. Naphtholate AS (GrE) is a mixture of Naphthol AS and a soluble oil. Brenthol H soluble 50% paste (British Synthetics, Ltd.) (S) is an aqueous paste of a readily soluble combination of Naphthol AS and pyridine. Baths prepared from it can be kept almost indefinitely without occurrence of oxidation or polymerisation. Making use of the table of

melting points (*cf.* B., 1924, 704) the following commercial bases have been identified: Fast Garnet base CL (JWL), Fast Garnet base L (JWL), Fast Garnet GB base (GrE), and Fast Garnet GC base (GrE), are *o*-aminoazotoluene. Fast Red base GL (JWL) and Fast Red GL base (GrE) are 3-nitro-*p*-toluidine. Fast Red base GL (JWL) and Fast Red GL base (GrE) are 3-nitro-*p*-toluidine. Fast Red base RL (JWL) and Fast Red RL base (GrE) are 5-nitro-*o*-toluidine. Fast Scarlet GL base special (JWL) and Fast Scarlet G base (GrE) are 4-nitro-*o*-toluidine. Fast Scarlet Base GCL (JWL) and Fast Scarlet GC base (GrE) are 4-nitro-*o*-toluidine hydrochloride. Fast Scarlet base 2 GL (JWL) and Fast Scarlet GG base (GrE) are 2:5-dichloroaniline. Fast Yellow base GL (JWL) and Fast Yellow G base (GrE) are *o*-chloroaniline, whilst Fast Yellow base GCL (JWL) and Fast Yellow GC base (GrE) are the hydrochloride of *o*-chloroaniline. Fast Orange G base (GrE) is *m*-chloroaniline. Fast Red KB base (GrE) used for colours fast to kier boiling is 4-chloro-*o*-toluidine. The Rapid Fast Printing Colours (GrE) contain as one component the stable nitrosoamine of a diazotised amine in a faintly alkaline medium. The Fast Salt series (GrE) are stabilised diazo-compounds which form solutions acid to litmus and can be used directly for the development of insoluble azo colours on prepared calico. Fast Red Salt B (GrE) is diazotised 5-nitro-2-aminoanisole, Fast Red Salt GG (GrE) is diazotised 2:5-dichloroaniline, Fast Red Salt GL (GrE) is diazotised 3-nitro-*p*-toluidine, and Fast Scarlet Salt R (GrE) is diazotised 4-nitro-2-aminoanisole. Permanent Red 2G (AGFA) and Monolite Red 2G (BDC) are diazotised *m*-dinitroaniline coupled with *β*-naphthol.

A. COULTHARD.

PATENTS.

Manufacture of finely subdivided pigment dyes. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 240,852, 1.10.25. Conv., 1.10.24. Not yet accepted).—The fine subdivision of organic or inorganic dyes or pigments in aqueous suspension is retained on drying by intimately mixing with a resin or a solid fatty acid, for example by mixing an alkaline or other solution of the resin or fatty acid with the colour suspension and precipitating by acid or other precipitant, or alternatively by fusing the materials together or by grinding them in a colloid mill. The preparations may be used as such or the admixed substance may be eliminated before use by means of solvents without destroying the subdivision. For example a 20% aqueous paste of a dyestuff, such as that obtained by coupling diazotised *m*-nitro-*p*-toluidine with acetoacetanilide, is well stirred with an alkaline solution of a fatty acid, colophony, copal, or an alkali-soluble artificial resin, and the resulting mixture is acidified with hydrochloric acid, filtered, washed, and dried.

Manufacture of azo dyes. BRITISH DYESTUFFS CORP., LTD., K. H. SAUNDERS, and H. GOODWIN (E.P. 242,061, 22.10.24).—Azo dyestuffs yielding fast pink to bluish-red shades on printing with chrome mordants on calico, are obtained by coupling diazotised 1-amino-2-hydroxy-3-carboxynaphthalene-6-sulphonic acid with pyrazolones, preferably those containing one or more carboxyl groups, such as 3′-carboxy-1-phenyl-3-methyl-5-pyrazolone, 3′-oxamidophenyl-3-methyl-5-pyrazolone, and 3′-carboxyphenyl-3-carboxy-5-pyrazolone. 1-Amino-2-hydroxy-3-carboxynaphthalene-6-sulphonic acid is obtained by sulphonating *β*-hydroxynaphthoic acid, coupling the product with azobenzene, and reducing the azo compound.

L. A. COLES.

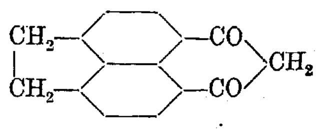
Orthohydroxyazo dye. O. KALTWASSER, H. KIRCHHOFF, and H. OEHRN, Assrs. to A.-G. F. ANILIN-FABR. (U.S.P. 1,556,329, 6.10.25. Appl., 15.5.25).—A sulphonated *o*-hydroxy-aromatic amine is diazotised and coupled with an unsulphonated 1-naphthyl-3-methyl-5-pyrazolone to yield wool dyestuffs, the after-chromed tints of which, and those obtained by dyeing in the presence of a chrome-mordant, are orange to bluish-red and are fast to light and stoving. For example 1-*β*-naphthyl-3-methyl-5-pyrazolone is coupled with 4-nitro-2-aminophenol-6-sulphonic acid. The product in presence of a chrome-mordant gives orange tints on wool.

T. S. WHEELER.

Production of dyestuffs [from glycosine]. K. LEHMSTEDT (G.P. 414,962, 3.8.22).—The *nitro-compound*, $C_6H_5O_{10}N_7$, obtained by nitrating glycosine (di-iminazolyl), is treated with reducing agents. The dyestuff, $C_6H_5O_7N_6,2NH_3$, obtained using concentrated ammonium sulphide as reducing agent, yields on wool and silk yellowish-brown shades, and that obtained using sodium hyposulphite yields on wool grey to black shades.

L. A. COLES.

Manufacture of 1:4:5:8-naphthalenetetracarboxylic acid and its derivatives. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 240,859, 2.10.25. Conv., 2.10.24. Not yet accepted).—Naphthalene-1:4:5:8-tetracarboxylic acid and its derivatives are obtained by condensing acenaphthene, or its derivatives having free *peri*-positions, with malonyl chloride and oxidising the *peri*-acenaphtheneindandiones so obtained. For example acenaphthene is condensed with malonyl chloride in presence of carbon disulphide and aluminium chloride, and the resulting *peri*-acenaphtheneindandione of the formula



oxidised with alkaline permanganate to naphthalene-1:4:5:8-tetracarboxylic acid.

Production of vat [perylene] dyestuffs. KALLE U. CO. A.-G., Assees. of M. SCHMIDT and W. NEUGEBAUER (G.P. 415,711, 2.5.22. Addn., to 386,057).—The dyestuffs are obtained by treating perylenetetracarboxylic acid monoimide or its derivatives (*cf.* E.P. 201,786; J., 1923, 968 A) with ammonia or with primary aliphatic or aromatic amines. For example, the dyestuffs obtained by heating the

monoimide with aniline and with *p*-chloroaniline yield on cotton from the vat red shades fast to chlorine, and bluish-red shades, respectively. L. A. COLES.

Production of β-phenyl-β-hydroxypropiono-*o*-carboxylic anhydrides. CHEM. FABR. VORM. WEILER-TER MEER (G.P. 416,073, 13.12.23).—The compounds, which are of use as intermediates in the manufacture of dyestuffs and drugs, are produced by the action of sulphuric acid upon *o*-cyanocinnamic acid or its derivatives (*cf.* G.P. 411,955; B., 1925, 693). For example, *o*-cyanocinnamic acid, m.p. 137°, is boiled for 5 hrs. with 30% sulphuric acid; *benzhydrylaceto*-o-*carboxylic anhydride* (*β-phenyl-β-hydroxypropiono*-o-*carboxylic anhydride*), m.p. 148—149°, crystallises out on cooling. The isomeric *o*-cyanocinnamic acid, m.p. 254°, remains unchanged during the treatment, and if mineral acids other than sulphuric acid be used, the acid of m.p. 137° is transformed into the isomeride of m.p. 254°. 4-Hydroxy-2-cyanocinnamic acid (*loc. cit.*) is converted into 4-*hydroxybenzhydrylaceto*-2-*carboxylic acid*, m.p. 220—222°, by boiling with hydrochloric acid, *d* 1·162, for 6 hrs. under a reflux condenser. L. A. COLES.

Production of dihalogendiphenylmethanedicarboxylic acids. H. WEIL (G.P. 416,544, 6.1.23).—The compounds are produced by the condensation below 40° of *o*- and *p*-halogenbenzoic acids with formaldehyde or compounds yielding it. For example, trioxymethylene is stirred with a solution of *o*-chlorobenzoic acid in concentrated sulphuric acid at 15° until it has dissolved. After keeping for 72 hrs., the solution is poured into water, and uncombined *o*-chlorobenzoic acid is removed by boiling. The *dichlorodiphenylmethanedicarboxylic acid* formed has m.p. 274°, and the *isomeride* produced from *p*-chlorobenzoic acid has m.p above 300°. The products are of use in the manufacture of dyestuffs and drugs. L. A. COLES.

Manufacture of [triarylmethane-azo] dyestuffs containing chromium. SOC. OF CHEM. IND. IN BASLE (E.P. 231,446, 2.3.25. Conv., 28.3.24).—See U.S.P. 1,551,073; B., 1925, 875.

Manufacture of [azo] dyestuffs. O. Y. IMRAY. From SOC. OF CHEM. IND. IN BASLE (E.P. 242,867, 30.3.25).—See U.S.P. 1,549,901; B., 1925, 875.

Azo dyestuff. R. STÜSSER, Assr. to FARBENFABR. VORM. F. BAYER U. CO. (U.S.P. 1,560,949, 10.11.25. Appl., 15.4.24).—See E.P. 215,769; B. 1925, 623.

4-Hydroxynaphthalene 1-aryl ketones. G. DE MONTMOLLIN, E. REBER, G. BONHÔTE, and J. SPIELER, Assrs. to SOC. OF CHEM. IND. IN BASLE (U.S.P. 1,552,472, 8.9.25. Appl., 30.1.23).—See G.P. 378,909; J., 1923, 1123 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Retting of flax with *Plectridium pectinovorum* (*Bac. amylobacter*, A.M. et Bredemann) and *Bacillus felsineus*, Carbone. G. RUSCHMANN and W. BAVENDAMM (Zentr. Bakt. u. Parasitenk., Abt. II., 1925, **65**, 43—58; from Chem. Zentr., 1925, II., 1687; *cf.* B., 1925, 876).—New evidence is brought forward that in the retting process as carried out in Germany, Carbone's bacillus takes no part in the fermentation of pectin and, even if used in pure culture, succumbs to the competition of the amylobacteria. Possible causes of this difference between German and Italian retting are discussed. C. T. GIMINGHAM.

Comparison between bamboo cellulose and cotton cellulose. G. KITA and K. AZAMI (Cellulose Ind., Tokyo, 1925, **1**, 162–164).—The maximum yield of cellobiose octa-acetate by Klein's method (*cf.* J., 1912, 713) obtained from bamboo cellulose prepared by chlorination was 70·82% (m.p. 220°), corresponding with 33·84% of cellobiose, after acetolysis for 7 days. The yield of cupric-reducing sugar obtained by Ost's method of total hydrolysis (*cf.* J., 1910, 688) corresponded with 97·5% of the theoretical value calculated as dextrose, and 90·4% of the theoretical quantity of crystallised dextrose was recovered. Hence the constitution of bamboo cellulose is similar to that of cotton cellulose. J. F. BRIGGS.

Constituents of bamboo. K. AZAMI and S. SENGOKU (Cellulose Ind., Tokyo, 1925, **1**, 157—161).—Analysis of raw bamboo stems including knots showed for a one-year old stem: Cellulose 51·7% and lignin 25·05% in the upper portion, 52·0 and 25·06% in the middle, 52·0 and 29·22% respectively in the lower portion. A three-year old stem showed similarly 52·15 and 26·05% in the upper portion, 53·1 and 28·26% in the middle, and 53·4 and 26·63% in the lower. The figures are expressed on dry weights and those for the cellulose on material previously extracted with benzene. Pentosan (xylan) ranged between 18 and 20%. Values for ash ranged from 1·65 to 2·02%; benzene extract, from 0·19 to 0·55%; alcohol extract, from 2·59 to 5·26%, the values for these extracts being highest in the upper portion of three-year old stems. Pectin varied from 0·0008 to 0·0360% and methyl value from 2·616 to 2·841%. J. F. BRIGGS.

Action of highly concentrated hydrochloric acid on cellulose.—K. ATSUKI (Cellulose Ind., Tokyo, 1925, **1**, 53—61).—Cotton cellulose was treated at 10° with 100 times its weight of hydrochloric acid at 40, 41, 43, and 45% strength, and the course of the hydrolysis was followed by measurements of viscosity and specific rotatory power. The increase in rotation is more rapid the higher the concentration of the acid. The rotation–time curves show two points of deflection, the first after about 5 hrs. with 45% acid or 10 hrs. with 40% acid and the second after 15 hrs. with 45% acid or 30 hrs. with 40%. The viscosity falls rapidly along the first step of the specific rotation curve and remains constant afterwards. The addition of 1% of zinc chloride or 0·5% of sodium chloride to the acid retards the solution of the cellulose and inhibits to some extent the hydrolysis. The addition of 0·5% of calcium

chloride suppresses the solvent action of the hydrochloric acid, the cellulose being disintegrated to a powdery form after a few days. The solution of cellulose is considered to take place by its association with the hydrochloric acid in virtue of its OH-groups reacting as water so long as the concentration of the acid is such that it is still unsaturated with water of hydration; dispersion of the cellulose under these conditions is progressive. The end product of the hydrolysis is dextrose, the intermediate products being colloids and then crystalloids in stages. By precipitation at an early stage after solution the cellulose is obtained in a modified form very highly hydrated. It is physically unsuitable for nitration or acetylation, but can be centrifuged and converted into viscose. J. F. BRIGGS.

Factors in the caustic soda absorption of cellulose and their effects on the resulting viscose. M. NUMA (Cellulose Ind., Tokyo, 1925, 1, 87—101).—In the preparation of alkali-cellulose for viscose, the optimum results as regards the viscosity of the viscose solution and the physical quality of the regenerated cellulose are obtained when the steeping is performed under the following conditions: concentration of sodium hydroxide, 15%; temperature of steeping bath, 20°; time of immersion 24 hrs. The surface area of the sample of cellulose influences the absorption of sodium hydroxide, maximum absorption being obtained with pieces of 0·5—1 sq. cm.; stirring does not influence the absorption. The absorption of sodium hydroxide is increased by the addition of sodium salts, the carbonate having the most pronounced effect and the sulphate the least; the bicarbonate and the phosphate cause a decrease in the absorption. It is suggested that the cellulose does not absorb the undissociated sodium hydroxide molecules but the sodium ion. J. F. BRIGGS.

Determination of moisture in fibrous materials. C. G. SCHWALBE (Ver. Zellstoff- u. Papier-Chem., Hauptversamml., 1924, 121—123).—The usual method for the determination of moisture is by heating in a weighing bottle at 105°. Care must be taken that the temperature is equalised in all parts of the oven, and the weighing bottles should not be directly in contact with the metal shelves of the oven. Good exposure of the loosely packed material should be ensured. This method is not accurate in the case of materials containing soluble carbohydrates, such as pectin, or those which are liable to lose volatile constituents other than water, such as resinous woods. The time required is 4—8 hrs. By the use of a vacuum weighing-bottle the time may be shortened and the temperature of the water-oven will suffice. With suitable apparatus the use of vacuum may be further supported by the presence of phosphorus pentoxide. The method of distillation in the presence of a hydrocarbon gives results of sufficient accuracy; it is particularly suitable for special cases, *e.g.*, where large samples must be taken or where resinous or fatty materials have to be dealt with. Obermiller recommends for the drying of large bulk samples a water-jacketed oven at 95—100°, a current of air of normal dryness, derived from a source free from the products of combustion, being circulated through the material. This is particularly desirable in the drying of compact materials such as wood pulp. J. F. BRIGGS.

Ungerer's system for digesting wood by the soda process. JENKE (Ver. Zellstoff- u. Papier-Chem., Hauptversamml., 1924, 177—180).—Ungerer's process is still being worked at the Stuppach pulp mill with a few modifications. The digester plant consists of a battery of 9 well-insulated digesters inter-connected both top and bottom, of which 5 constitute a working series, while the others are in course of charging and discharging. The digesters have a capacity of 2·07 cb.m. of wood. The caustic liquor (5·6—6·0% of sodium hydroxide) is contained in a furnace-heated boiler consisting of a tubular preheater and horizontal cylindrical body, which are directly under fire, and an upper cylindrical reservoir, containing both liquor and steam, not directly exposed to the furnace gases. The liquor is forced under a steam pressure of 6·6—8 atm. from this reservoir through the battery of digesters in such a way that the fresh liquor enters the digester which is next ready to be discharged and the most exhausted liquor enters the digester most recently filled. As a digester charge of spent liquor is drawn off for transfer to the recovery plant, the liquor in each of the other digesters is passed one stage forward, while the digester ready for discharge is drained under the steam pressure from the liquor boiler. The pulp from this digester, saturated with practically clean fresh liquor, is conveyed to a system of Shanks' lixiviating tanks, the ultimate washings from which are returned to the caustic liquor plant. Thus, no washings are treated in the soda-recovery system. The 9 digesters give 24 charges per 24 hours. The make-up in the soda-recovery plant consists partly of sodium sulphate and partly of carbonate; excess of sulphate in the make-up causes corrosion in the liquor preheater. The consumption of chemicals per 100 kg. of cellulose is 4·04 kg. of sodium carbonate, 7·08 of Glauber's salt, and 25·6 kg. of quicklime. J. F. BRIGGS.

Reducing power of sulphite-cellulose waste liquor. KOTIBHASKER.—See VI.

Linoleum and triolin. SIMON.—See XIII.

PATENTS.

Degreasing raw wool. A. M. BRUCKHOFF (E.P. 241,314, 28.7.24).—Raw wool containing 10—30% of natural fat is degreased by extraction with warm (not boiling) acetone, the resulting wool having a fat content of 2—3%. Degreasing may be effected by successive extraction of wool with 3—5 times its weight of cold acetone or by passing the wool successively during 15 mins. through a series of extraction chambers containing acetone, the temperature in the last chamber preferably being 30—40°. The fats extracted by cold and warm (30—40°) acetone have m.p. 15—25° and 60°, iodine values 22—28 and 10, and saponification

values 108—115 and 70 respectively. It is advantageous to dry the wool till it contains 2—3% of moisture before degreasing. The solubilities of wool wax and those constituents of wool fat having a low melting point are increased six- and two-fold respectively when the temperature of the acetone used for extraction is raised by 25°; the residual fat content of the extracted wool is controlled by the temperature of extraction. A. J. HALL.

Manufacture of cellulose acetate. L. A. LEVY (E.P. 240,624, 9.4.24).—Cellulose acetate soluble in acetone is rapidly prepared by treating cellulose (containing 6—7% of moisture) with a mixture containing acetic acid, acetic anhydride, a condensing agent, such as sulphuric acid, and a catalyst, such as the acetate or sulphate of vanadium, nickel, cobalt, or chromium. For example, 2·5 lb. of cellulose are kneaded at a temperature not exceeding 15° with a mixture containing 5 lb. of acetic acid, 5 lb. of acetic anhydride, 0·2 lb. of sulphuric acid, and 2·5 lb. of chromium acetate until a transparent, viscous liquid free from fibres is obtained, the mixture being then maintained at 30° until the resulting cellulose acetate is freely soluble in acetone; the preparation is complete within 18 hrs. Salts of magnesium, aluminium, sodium, antimony, uranium, manganese, cerium, tin, niobium, and thorium are not satisfactory catalysts. A. J. HALL.

Treating cellulose acetate. E. S. FARROW, JUN., Assr. to EASTMAN KODAK Co. (U.S.P. 1,557,147, 13.10.25. Appl., 20.1.25).—Cellulose acetate (soluble in chloroform) is dissolved in a solution of phosphoric acid and the partially hydrolysed. A. J. HALL.

Preparation of flocculent cellulose products. WOLFF U. Co., Assees. of E. CZAPEK and R. WEINGAND (G.P. 415,588, 17.6.22).—Wood-pulp, paper, and similar fibrous materials are mechanically disintegrated in the presence of dispersing (not swelling) agents, such as ether, alcohol, and chloro-derivatives of acetylene, under such conditions that no decomposition of the fibres occurs. The soft products thereby obtained readily absorb acids, bases, and salts, and are particularly suitable for the manufacture of nitrocellulose, wadding, and viscose, yielding solutions of the last-named which are very easily filtered. A. J. HALL.

Production of resin (from pulp mill black liquor). F. E. GREENWOOD, Assr. to PINE WASTE PRODUCTS, INC. (U.S.P. 1,560,420, 3.11.25. Appl., 25.8.20).—The resinous components of pulp-mill black liquor are treated with an acid which will liberate the resin and a solvent which will remove it from the aqueous liquor. The resin solution is then treated with a second solvent, immiscible with the first, to remove the resin from the accompanying impurities. A. R. POWELL.

Utilisation of waste material from de-inking of paper in making paper. L. E. GRANTON (E.P. 240,924, 10.7.24).—Waste fibrous material (usually containing particles of lampblack) obtained by well known methods from the water used for washing de-inked printed paper (*cf.* E.P. 222,160; B., 1924, 977) is mixed with other paper pulp and suitable filling agents, such as china clay and rubber latex, and then converted into paper which is suitable for wrappers, handbills, and tickets. The strength of the resulting paper is not materially diminished if its content of wastage does not exceed 30%, although papers containing 60% of wastage may be prepared (*cf.* G.P. 411,334; B., 1925, 842). A. J. HALL.

Sizing paper. J. JANSER (G.P. 415,675, 30.11.22. Conv., 7.2.22).—Paper is sized with 8—20% of a colloidal or highly dispersed solution (prepared by means of a colloid mill or similar apparatus) of wood or other cellulosic material. The pores of the paper are filled and it acquires transparency and a high tensile strength. A. J. HALL.

Apparatus for removing deposited impurities from textile or metallic fabrics, braided or felted materials used in the manufacture of paper. W. FUHRMANN (G.P. 416,281, 14.3.23).—The materials are subjected to electro-osmosis while maintained stationary or moving between moving or stationary electrodes in a washing trough, the impurities being removed and deposited on the anode by cataphoresis. A. J. HALL.

Gutter or channel for treating artificial threads or yarns, particularly imitation-silk yarns, with acids or an acid-containing liquor. E. ELSAESSER and W. ZUR LÖWEN, Assrs. to AMER. BEMBERG CORP. (U.S.P. 1,561,445, 10.11.25. Appl., 29.10.23).—See E.P. 209,376; B., 1924, 251.

Production of [mechanical] wood-pulp. C. J. STERNKOPF (E.P. 234,120, 14.5.25. Conv., 15.5.24).

Converting methyl sulphide into carbon tetrachloride etc. (G.P. 416,603).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Reducing power of sodium hyposulphite in alcoholic solution [and its use for bleaching silk]. J. POKORNY (Rev. gén. Mat. col., 1925, **29**, 288).—The increased reducing power of sodium hyposulphite in aqueous alcoholic solution previously described (*cf.* B., 1925, 127) may be used for bleaching natural silk. Air-dry silk is thoroughly bleached (cotton dyed with indigo is simultaneously decolorised so that its colour is not restored by oxidation with a dichromate) by immersion for ¾ hr. in a mixture containing 100 g. of sodium hyposulphite, 2 litres of denatured alcohol, and 600 c.c. of water, and further quantities of silk may be bleached afterwards in the remaining liquor after successive additions of suitable quantities of water (about 300 c.c.) until the sodium hyposulphite has completely dissolved. A yellow acidic substance having an agreeable odour and soluble in water is obtained by evaporation of the spent bleaching liquor, and it is re-precipitated from its aqueous solution by addition of ammonia or hydrochloric acid. Copper

is stained black rapidly and strongly by immersion in an aqueous solution of sodium hyposulphite, but only slowly in a similar solution containing alcohol.
A. J. HALL.

Reducing power of sulphite[-cellulose] waste liquor, and its use in dyeing and bleaching. M. G. KOTIBHASKER (J. Soc. Dyers and Col., 1925, 41, 361—362).—The waste liquor used was that left after digesting rasped Scotch fir for 14—16 hrs. at 160° in 6 times its weight of a solution containing 1·4% CaO and 4·5% SO_2. The final liquor had *d* 1·050, and contained 9·9% of total solids (CaO 0·63%). The liquor produces a satisfactory indigo vat, 17·4–20 c.c. of it being equal to 1 g. of hyposulphite in reducing power. Calico can be dyed very evenly in such a vat. Fabrics dyed with indigo can be stripped by immersion in a warm bath of the waste liquor and sodium hydroxide and the indigo regenerated and recovered to the extent of 50—63%. Indanthrene, Flavanthrene, and Sulphur Black are reduced by the waste liquor, but with Para Red, reduction is only partial.
A. COULTHARD.

Products used for insoluble azo colours. ROWE and LEVIN.—See IV.

PATENTS.

Bleaching wool. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of L. LÖCHNER (G.P. 415,583, 1.8.23).—Wool is bleached by treating it for 2—5 mins. with a cold or warm solution of sodium bisulphite of 0·5—1·0° B. (*d* 1·003—1·007), then removing excess of the solution, and drying.
A. J. HALL.

Bleaching bristles. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of L. LÖCHNER and G. KÖHLER (G.P. 417,407, 25.6.24).—The usual bleaching treatment with peroxides is preceded by treatment of the bristles with a solution containing free chlorine (*e.g.*, an acidified solution of bleaching powder or sodium hypochlorite, or chlorine water), the bleaching process being thereby shortened and an improved white colour obtained.
A. J. HALL.

Treatment of cellulosic materials, fibres, yarns, and fabrics with liquids [sulphuric acid]. KNOW MILL PRINTING CO., LTD., T. L. MORT, and F. W. WEEKS (E.P. 241,246, 20.5.24).—Pattern effects on cotton, linen, and similar material are obtained by printing or spraying it with a mixture containing sulphuric acid and methyl alcohol, acetone, or acetic acid or their homologues, these latter substances having a retarding effect on the rapidity but not effectiveness of sulphuric acid on cellulose. For example, transparent effects accompanied by negligible decrease of strength are obtained by treating cotton fabric with a mixture containing 25 g. of acetic acid and 75 g. of sulphuric acid at 15° for 30 secs. or with a mixture containing 46 g. of alcohol and 196 g. of sulphuric acid at 15° for 60 secs. The treatment may be preceded or followed by mercerisation or calendering.
A. J. HALL.

Dyeing animal fibres and fabrics of a protein nature. S. W. WILKINSON (E.P. 242,027, 25.6.25).—After exposure to the simultaneous action of ozone and ammonia, such materials as wool, fur, bristles, silk, leather, and feathers have an increased affinity for all classes of dyestuffs and the resulting dyeings have greater fastness to light, washing, and acids. A satisfactory method of treatment consists of impregnating wool or similar material with a 5% solution of ammonia, removing excess of liquor by hydro-extraction, and then exposing it for several hours to a current of moist ozonised air containing 1 pt. of ozone per 1000 pts. of air, subsequently dyeing it or drying it at a low temperature (the increased affinity for dyestuffs is retained for a considerable time). Acidity produced during the treatment with ozone is neutralised by repeated treatment of the material with ammonia. White furs sometimes acquire a silky lustre when subjected to the treatment described above.
A. J. HALL.

Dyeing leather. KALLE U. CO. A.-G., Assees. of K. LUCK and R. ZAHN (G.P. 417,209, 28.5.24).—Leather is dyed with acid or direct dyestuffs in the presence of salts of metals such as copper, zinc, iron, aluminium, and titanium, and of substances which hinder the precipitation in the dye-bath of compounds of the metal with the dyestuff, suitable substances including lactates, tartrates, and sulphite-cellulose waste liquor.
L. A. COLES.

Drying, lustring, and finishing machines for fabrics. CALICO PRINTERS' ASSOC., LTD., and F. FARNWORTH (E.P. 242,749, 10.10.24).

Dyeing machinery. J. DEAN (E.P. 242,790, 15.11.24).

Apparatus for treating vegetable, animal, or artificial fibres with dyes or other liquids. J. SCHLUMPF (E.P. 242,857, 14.3.25).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Chemistry of the lead chamber process [of making sulphuric acid]. F. RASCHIG (Z. angew. Chem., 1925, 38, 1001—1010).—Reprint of the author's paper published in this J., 1911, 166.
C. IRWIN.

Mechanical condition of ammonium sulphate. A. D. CUMMINGS (Gas J., 1925, 172, 357—359).—Ammonium sulphate may be discoloured by the black slime which accumulates in saturators. The insoluble portion of a typical sample contained 35·0% of copper sulphide, 12·2% of iron, 30·1% of carbon, and 10·1% of tar. The copper was derived from the centrifugal basket. The caking of dry salt may be minimised by the elimination of free sulphuric acid, lime, soda, free ammonia, and pyridine, by operating the saturator so as to produce uniform crystals of larger size, by the adoption of moderate drying temperatures, and by the elimination of dust.
H. HOLLINGS.

Control of electric furnaces. SCHLUMBERGER.—See XI.

Iron compounds. MILLER.—See XXIII.

PATENTS.

[Sulphur dioxide for] sulphuric acid process. GRASSELLI CHEMICAL CO., Assees. of J. C. BOERTLEIN (E.P. 240,801, 19.8.25. Conv., 4.10.24. Not yet accepted).—Sulphur dioxide is produced by using sulphur as fuel in an internal-combustion engine, which may be of the Diesel type. The exhaust gases may be passed into a chamber or burner in which any residual sulphur is oxidised, and the sulphur dioxide, after purification, if this be necessary, and possibly adjusting its temperature, may be converted into sulphuric acid. To avoid corrosion the air supplied to the engine is dried, and the sulphur may be introduced into the cylinder after being melted by means of superheated steam or by the heat of the combustion gases. The melting pot may be let into a wall of the combustion chamber in which the residual sulphur is burned.

Method of manufacturing sulphuric acid. J. V. SKOGLUND (U.S.P. 1,559,292, 27.10.25. Appl., 25.3.24).—In the lead chamber process for the manufacture of sulphuric acid, the gases leaving the last chamber pass in succession through a small preliminary Gay-Lussac tower, a water scrubber, and a final Gay-Lussac tower. Nitrogen peroxide is decomposed by the water, yielding nitric acid in solution and nitric oxide. The latter, after re-oxidation, is absorbed in the last tower. C. IRWIN.

Production of sulphuric acid in towers. R. VETTERLEIN, and CHEM. FABR. ZU SCHÖNINGEN (G.P. 416,859, 29.11.24).—Apparatus for the manufacture of sulphuric acid comprises a Glover tower and one or more reaction towers packed with filling material, in which both oxidation and absorption take place. The reaction towers are situated close to one another, and their total cross-sectional area is many times that of the Glover tower. L. A. COLES.

Producing soluble carbonates or hydroxides from insoluble carbonates, oxides, or hydroxides. A. F. MEYERHOFER, Assee. of E. DE HÄEN A.-G. (E.P. 219,971, 24.7.24. Conv., 2.8.23).—To prepare for example sodium carbonate from calcium carbonate, the latter is stirred in water for some hours with sodium fluoride. The solution is filtered from the precipitate of calcium fluoride, the latter washed, and the solution and washings are evaporated. The calcium fluoride is dissolved in dilute hydrochloric acid and heated with sodium chloride and silicon fluoride, yielding sodium silicofluoride. This is decomposed by heating into sodium fluoride and silicon fluoride, which are returned to the cycle. C. IRWIN.

Process of making alkali [sodium] carbonate. G. N. LIBBY, Assr. to NAT. MAGNESIA MANUF. CO. (U.S.P. 1,558,901, 27.10.25. Appl., 25.5.24).—Pulverised magnesia is added to a saturated solution of natural soda (trona). H. ROYAL-DAWSON.

Separating hafnium and zirconium. N. V. PHILIPS GLOEILAMPENFABR., Assees. of D. COSTER and G. HEVESY (E.P. 219,983, 29.7.24. Conv., 30.7.23).—The basic salts of zirconium and hafnium, in which the basic radical contains more oxygen than ZrO or HfO, are separated by fractional crystallisation. Thus a mixture of the oxychlorides, $Zr_2O_3Cl_2$, $5H_2O$ and $Hf_2O_3Cl_2$, $5H_2O$ (in the precipitation of which iron, aluminium, and the like remain in the mother liquor) is dissolved in 50 pts. by weight of alcohol, and 125 pts. by weight of ether are added. The zirconium becomes concentrated in the precipitate so produced and the hafnium in the mother liquor. Acetone may also be used as precipitant. C. IRWIN.

Production of aluminium chloride and alumina. CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 240,834, 29.9.25. Conv., 2.10.24. Addn. to 205,563. Not yet accepted).—The ferruginous liquors obtained in carrying out the process described in the chief patent (J., 1923, 1222 A) are diluted and treated with aluminous material such as burnt clay to remove most of the iron and enrich the liquors in aluminium chloride. The liquors can then be evaporated as in the process of the chief patent, and the clay is washed with water to remove the iron hydroxide, after which the clay may be used with other raw material in the process.

Catalytic synthesis of ammonia. S. G. S. DICKER. From H. HARTER and A. T. OTTO (E.P. 241,771, 21.1.25).—Two catalyst chambers are used, the inner one working at high pressure, *e.g.*, 500 atm., the outer one in the form of a jacket or a coiled tube at a lower pressure, *e.g.*, 300 atm., the gas stream being divided between the two. The inner chamber contains the usual heating elements, the outer one being heated by conduction only. The continuity of the process is secured by regulating the pressure in the inner chamber or the relative gas velocities in the two. The heat of reaction at the higher pressure is thus economically used, whilst the structural difficulties are reduced by the partly counter-balancing outside pressure. The ammonia produced can be liquefied by expansion only. C. IRWIN.

Catalytic apparatus for the synthesis of ammonia. SYNTHETIC AMMONIA AND NITRATES, LTD., and F. H. BRAMWELL (E.P. 241,817, 4.5.25).—The catalyst chamber is surrounded by a heat interchanger consisting of two coaxial tubes wound into two spirals. The exit gases pass through the inner tube and the entering gases through the outer annulus. The remainder of the space outside the catalyst chamber is tightly packed with asbestos or other lagging. When the inner chamber is at a temperature of 600° the temperature of the outer wall does not exceed 100°, and ordinary mild steel may be used for its construction. C. IRWIN.

Manufacture of titanium sulphate. H. N. MCCOY, Assr. to LINDSAY LIGHT CO. (U.S.P. 1,559,113, 27.10.25. Appl., 14.6.24).—Basic titanium sulphate is treated with fuming sulphuric acid to form the soluble sulphate. H. ROYAL-DAWSON.

Production of easily filtered, hydrated titanic acid.—DEUTSCHE GASGLÜHLICHT-AUER-GES. (G.P.

417,726, 26.4.22).—Hydrated titanic acid is precipitated by passing steam into heated solutions containing titanic salts, *e.g.*, titanic sulphate, the steam being superheated to such a temperature that on passage into the boiling solution, its temperature falls at least 10°. L. A. Coles.

Elimination of iron from solutions of leucitic rocks. U. Pomilio and F. Giordani, Assrs. to Pomilio Bros. Corp. (U.S.P. 1,559,179, 27.10.25. Appl., 25.8.22).—Leucitic rock is treated with acid and after removing the silica, the solution is subjected to electrolysis to precipitate the iron. H. Royal-Dawson.

Producing alkali xanthates. R. B. Crowell and G. F. Breckenridge, Assrs. to Western Industries Co. (U.S.P. 1,559,504, 27.10.25. Appl., 18.5.25).—Alkali xanthates are produced by interaction of alkali hydroxide, alcohol, and carbon disulphide, in a liquid hydrocarbon medium which is chemically inert to the remaining ingredients. H. Royal-Dawson.

Manufacture of crystallised zinc sulphide. G. de Hédouville and P. Pipereaut (U.S.P. 1,560,380, 3.11.25. Appl., 2.1.24).—Solid oxidised zinc compounds are treated with hydrogen sulphide at a temperature between 250° and a red heat. A. R. Powell.

Production of strontium oxide from strontium carbonate. Chem. Fabr. Coswig-Anhalt G.m.b.H., and W. von Dieterich (G.P. 417,019, 23.4.24. Addn. to 396,214).—The process described in the chief patent (*cf.* E.P. 191,215; J., 1923, 451a) is used for the production of high-percentage strontium oxide. L. A. Coles.

Production of pure lithium compounds from minerals containing lithium, especially from lithia mica. Metallbank u. Metallurgische Ges., C. von Girsewald, and H. Weidmann (G.P. 417,169, 20.7.22. Addn. to 413,721).—In the process described in the chief patent (cf. B., 1925, 757), the lithium fluoride is decomposed by fusion with salts or compounds of metals, the fluorides of which are less soluble than lithium fluoride, *e.g.*, it is heated to fusion with calcium carbonate and a quantity of calcium oxide necessary for the formation of lithium oxide. L. A. Coles.

Extraction of crude barium sulphide and similar substances. P. Kircheisen (G.P. 417,339, 9.3.24).—The material is extracted in a closed rotating vessel provided with a perforated false bottom, so that the material can be extracted and subsequently washed with acid in the vessel. L. A. Coles.

Catalytic production of hydrogen. Synthetic Ammonia and Nitrates, Ltd., Assees. of W. Schultze (E.P. 220,649, 16.8.24. Conv., 16.8.23). —A catalyst of the ferric oxide type for the reaction $CO+H_2O=CO_2+H_2$ is prepared in a coherent form by the use of high pressure on the dry material without any binder. The raw material may be the precipitate produced by the addition of ammonia or of magnesium carbonate to a nitric acid solution of iron to which some potassium dichromate may be added. The precipitate is dried, ignited at 700°, and without screening, compressed into 0·5 g. discs in a Stokes tablet machine. These tablets do not disintegrate under the conditions of the reaction, are sufficiently porous to function efficiently as a catalyst, and can be handled without special precautions. C. Irwin.

Production of sulphuric acid. H. Klencke, Assr. to Amer. Lurgi Corp. (U.S.P. 1,561,985, 17.11.25. Appl., 28.3.24).—See G.P. 398,318; B., 1924, 942.

Producing hydrocyanic acid from sulphocyanic acid or its compounds. J. A. Du Bois (E.P. 214,999, 23.4.24).—See G.P. 410,418 and 411,104; B., 1925, 757.

See also pages 2, **Evaporating salt solutions** (E.P. 242,018). 5, **Purifying sulphided alkaline solutions** (E.P. 241,221); **Eliminating hydrogen sulphide from gases** (E.P. 241,248 and 241,252). 17, **Recovering cyanogen** (E.P. 241,669). 27, **Carbamide** (E.P. 241,123).

VIII.—GLASS; CERAMICS.

Colour imparted to glass by fluorine compounds. K. Fuwa (J. Jap. Ceram. Assoc., 1924, **32**, 280—294; *cf.* B., 1925, 99).—Cryolite, sodium silicofluoride, and sodium fluoride are approximately equally effective in producing opalescence in glass. Calcium fluoride is less effective. The milky colour is more easily produced in potash glasses than in soda glasses, and an increase in the content of alkalis favours the coloration. Glasses containing bivalent heavy metals are more susceptible to the coloration than those containing alkaline-earth metals. The author supports the view that the opalescence is due to colloidal silica produced by the action of fluorine compounds on silica; fluorides of alkaline-earth or other bivalent metals would, however, contribute to the effect in glasses containing these metals. Addition of arsenious oxide has no effect, and of potassium nitrate little effect on the action of fluorine compounds in producing opalescence. S. Kondo.

Colour imparted to glass by sulphur and its compounds. K. Fuwa (J. Jap. Ceram. Assoc., 1924, 32, 314—327).—Addition of sulphur generally produces a darker colour in potash glasses than in soda glasses. The batches containing sulphur foam remarkably, and the glasses are generally brittle. The colours produced by antimony pentasulphide are somewhat lighter than those given by sulphur. Glasses of the composition $K_2O,CaO,3SiO_2$, $K_2O,ZnO,3SiO_2$, and $K_2O,CaO,0{\cdot}5B_2O_3,3SiO_2$ were coloured a beautiful yellow by 1% of the sulphide, the glasses $R_2O,PbO,3SiO_2 + 1\%\ Sb_2S_5$ and $R_2O,PbO,0{\cdot}5B_2O_3,3SiO_2 + 0{\cdot}5$—$1\%\ Sb_2S_5$, formed light greenish alabasters; lead was reduced in the latter glasses. Potash and borosilicate glasses tend to produce alabasters on addition of cadmium sulphide. The glasses, K_2O,

$CaO,0{\cdot}5B_2O_3,3SiO_2$ + 5% CdS and $K_2O,MgO,0{\cdot}5B_2O_3,3SiO_2$ + 3–5% CdS are coloured a fine yellow. The colouring action of potassium sulphide is much weaker than that of sulphur. Generally speaking, the sulphides are entirely or partially decomposed at high temperatures, and the sulphur set free, or the products of its reaction with bases, impart to the glasses colours varying from yellow to dark red. Sulphates, if formed, may produce alabasters or opalescent glasses. The action of sulphur is reduced by addition of potassium nitrate, the effect of the latter being greater in soda-glasses than in potash-glasses. Potassium nitrate also tends to produce sulphates in potash glasses, and hence to form alabasters. Arsenious oxide acts in a similar way to potassium nitrate, except that it produces an alabaster very rarely. S. Kondo.

Colour imparted to glass by selenium. K. Fuwa (J. Jap. Ceram. Assoc., 1924, **32**, 327—342).—The colour given to glasses of the type $aR_2O,bRO,cSiO_2$ by elementary selenium is lighter when R_2O is Na_2O than when K_2O, and is brown in soda-glasses and pink or red in potash-glasses. Boric acid favours the development of pink. The colours of glasses of the composition $1{\cdot}3R_2O,RO,6SiO_2$ with 0·1% Se are: Na-Ca light brown, K-Ca dark red, Na-Mg brownish-pink, K-Mg light orange-pink, Na-Zn light greenish-blue, K-Zn orange, Na-Ba orange, K-Ba light pinkish-yellow, Na-Pb yellow, and K-Pb colourless. Potassium nitrate tends to counteract the effect of selenium, though its influence is slight. Potassium tartrate darkens the colour if RO is an alkaline earth, but has no effect when RO is a metal oxide. Arsenious oxide counteracts the effects of selenium. Soda-glasses are more susceptible to selenium than potash-glasses. S. Kondo.

Action of electrolytes on clays. S. Kondo (J. Soc. Chem. Ind. Japan, 1925, 1—10).—A theoretical paper in which an explanation of the author's results (*cf.* B., 1925, 849) is put forward based on the conception of an "ideal" clay as an ionisable kaolinic acid. Placticity is attributed to the mutual attraction of clay particles, which is opposed by the electrical repulsion set up as a direct consequence of the ionisation of kaolinates and kaolinic acid. Colloidal kaolinic acid produced by the hydrolysis of kaolinates and also by the action of acids on them plays an important rôle here, as it has enormous surface. S. Kondo.

Patents.

Plastic. W. Hoskins, Assr. to Economy Fuse and Manuf. Co. (U.S.P. 1,556,115, 6.10.25. Appl., 21.2.23).—A phosphate such as bone ash is mixed with quartz and treated with sulphuric acid to liberate phosphoric acid. On addition of calcium aluminate and water the aluminate acts on the phosphoric acid and rapid setting of the whole occurs. T. S. Wheeler.

Manufacture of articles from highly refractory materials. S. A. Myler (U.S.P. 1,559,275, 27.10.25. Appl., 7.10.24).—Graphite, pulverised soft coal, and petroleum oil are mixed to a putty, which is moulded to shape and heated to drive off the oil. The product is then dipped in tar and subsequently completely carbonised. C. Irwin.

Silica glass. L. B. Miller, Assr. to Gen. Electric Co. (U.S.P. 1,562,115, 17.11.25. Appl., 13.2.23).—See E.P. 189,926; J., 1923, 98 A.

Manufacture of chamotte bricks. S. E. Sieurin (U.S.P. 1,561,492, 17.11.25. Appl., 27.11.23).—See E.P. 218,495; B., 1924, 748.

IX.—BUILDING MATERIALS.

Curing concrete in a semi-arid climate. H. F. Gonnerman and C. L. MacKesson (Structural Materials Res. Lab., Lewis Inst., Chicago, Bull. 15, 1925. 37 pp.).—The best results from the point of view of strength and surface hardness for concrete cured in California (day temperature 30—35°, low humidity) are obtained by curing in wet earth. This maintains the moisture content of the concrete during the early stages of hydration about equal to the quantity of the original mixing water. There is no increase of strength when the curing is prolonged from 3 days to 7 or 14 days. Flake calcium chloride spread over the surface of the concrete does not give good results, as it absorbs water from the concrete itself, with corresponding loss of strength. Curing in air and with sodium silicate produced low strength and surface hardness; asphaltic paper was not so efficient as wet earth. The surface hardness of concrete measured by indentation methods is, in general, proportional to the transverse breaking strength. B. W. Clarke.

Patents.

Composition of matter hardened by metal chlorides. Granitite Manuf. Co., Assees. of H. J. Thompson (E.P. 228,847, 26.2.24. Conv., 6.2.24).—Calcined magnesite is mixed with powdered marble, fine silica sand, and finely ground quartz, with the addition of 6% by weight of the total of fibrous cellulose, such as sulphite pulp, wood pulp, etc. The mixture is gauged with magnesium chloride solution, which acts as a hardening agent. The material may be poured or pressed into moulds to form slabs, tiles, etc., which can be readily cut or nailed, and offer considerable resistance to buckling and warping. The mixture can also be applied as a plastering and possesses good thermal and electrical resistivity. B. W. Clarke.

Bituminous binding materials. L. S. van Westrum (E.P. 233,371, 8.11.23).—A bituminous waterproofing soap is prepared by saponifying a drying oil, preferably linseed oil, mixed with asphalt, by an alkali. Bitumen (30—50 pts.) is mixed hot with rosin (3—8 pts.) and linseed oil (3—8 pts.). Alkali lye (1 pt. of alkali to 3 pts. of water) is added, and mixing continued until saponification occurs. The product can be used for paving, briquette manufacture, etc. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 121,533.) T. A. Smith

Bituminous emulsions. H. A. Mackay (E.P. 233,430, 8.2.24).—A bituminous emulsion miscible with water is prepared by mixing with hot bitumen about 5% of an emulsifying agent containing cholesterol, and adding hot water to the mixture. Wool fat is a suitable emulsifying agent. The emulsion can be used in road making, briquette manufacture, etc. T. A. Smith.

Insulating cement or mortar. L. Caldwell, Assr. to Celite Co. (U.S.P. 1,556,488, 6.10.25) Appl., 29.1.23).—A heat-insulating cement or mortar is obtained by mixing 85—90 pts. of powdered diatomaceous earth with 10—15 pts. of an organic adhesive, such as glue or casein, adding 5% of a salt, such as sodium chloride or carbonate, or a mixture of such salts, and mixing with water. The inorganic salt increases the osmotic pressure and distends the adhesive, so that less of the latter is required. A preferred composition contains 83 pts. of diatomaceous earth, 10 of glue, 5 of sodium carbonate, 2 of aluminium sulphate, and 350 of water. T. S. Wheeler.

Manufacture of fused cement and sulphur dioxide. G. Polysius (G.P. 416,592, 13.9.24).—Raw materials containing sulphur are burnt, either alone or with suitable admixtures, so that a cement is produced which resembles aluminous cement in its properties, having a high initial strength and being resistant to chemical influences.
B. W. Clarke.

Producing cement. T. Fujiyama (U.S.P. 1,561,070, 10.11.25. Appl., 25.5.20).—See E.P. 168,406; J., 1920, 727 A.

Preservation of stone. A. P. Laurie (U.S.P. 1,561,988, 17.11.25. Appl., 8.4.24).—See E.P. 203,042; J., 1923, 1073 A.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Investigations on [cupola] burdens with briquettes of cast-iron and rusted and unrusted steel. O. Lechner (Stahl u. Eisen, 1925, 45, 1802—1809).—Additions of 5 to 30% of briquettes made of turnings of cast iron and of rusted and unrusted steel were added to a cupola charge of one-third of hæmatite and two-thirds of German No. 1 iron. With increasing addition of briquettes, the carbon and silicon content of the products decreased, the phosphorus remained constant, the sulphur increased, and the mechanical properties were improved. With the cast-iron briquettes the tensile strength reached a maximum with a 30% addition, the bending strength at 30%, the bending angle at 15%, and the impact value at 10%. With the rusted steel briquettes the properties named reached their maxima at 15%, 20%, 0%, and 15%, and with the clean steel briquettes at 25%, 20%, 20%, and 20% respectively. The fact that with the steel turnings the maxima did not occur at a 30% addition was due to the sulphur content of the product. The increase in sulphur was due to the great surface area of the turnings in contact with the coke. The cast-iron briquettes owing to their higher initial content took up less sulphur than the steel briquettes. In each series the Brinell hardness increased up to a 30% addition of briquettes. The machining properties were normal in all cases except for a 20% addition of rusted steel briquettes; in this case deposition of cementite made the metal difficult to machine. The addition of briquettes decreased the tendency to the formation of blowholes, and none of the samples solidified white. The metal made from clean steel briquettes was in general better than that obtained in the other cases. Photomicrographs showed that the tensile and impact test diagrams could be explained by the decrease in the amount of graphite, the appearance of cementite, the increase in pearlite, and decrease in ferrite in the structures throughout the series, in conjunction with the changes in the form of these constituents. The hardness curves were also explained by the hard constituents increasing throughout. The tests showed that in practice a 15% addition is the most advantageous, and that the mechanical properties can be deduced from the structure of the irons.
T. H. Burnham.

Constitution of iron. F. Wever (Physikal. Z., 1925, 26, 698—699; *cf.* B., 1925, 721).—A brief summary of our present knowledge of the allotropy of iron. The identity of the α and δ modifications of iron, first indicated by X-ray analysis, is confirmed by a study of the binary systems, iron–silicon and iron–tin. The solubility of carbon in γ-iron is regarded as due to the carbon atoms filling up the gaps in the face-centred lattice. The body-centred α-lattice cannot accommodate the carbon atoms without considerable distortion, which occurs when the solution in γ-iron is quickly cooled, and is the cause of the hardness of the quenched steel. A. B. Manning.

Influence of grain size on the magnetic properties of silicon iron sheet. O. von Auwers (Physikal. Z., 1925, 26, 699—700).—Investigation of the magnetic properties of a silicon-iron between 200° and 1000° gave optimum values between 700° and 1000°, depending on the atmosphere in which the specimen was heated. The recrystallisation, involving changes in grain size up to several thousand %, was independent of the atmosphere and occurred always between 900° and 1000°. These results do not accord with those of Yensen (J. Amer. Inst. Elect. Eng., May, 1924), who found a close correlation between watt-loss and grain size. A. B. Manning.

Recrystallisation of rolled silver sheet. R. Glocker, E. Kaupp, and H. Widmann (Z. Metallk., 1925, 17, 353—357).—In continuation and extension of earlier work (Glocker and Kaupp, B., 1924, 1016) the effect of annealing on the mechanical properties of silver sheet (99·7% Ag and 0·2% Cu) and on the grain growth has been studied with the help of X-rays. The minimum temperature at which recrystallisation takes place is a function not only of the ultimate reduction in thickness by rolling, but also of the reduction at each passage through the

rolls; in any case, however, *X*-ray photographs show no signs of recrystallisation having taken place below 200°. In rolled silver sheet the [011] planes of the crystallites are oriented parallel to the direction of rolling; after annealing at 200—700° the [113] planes are oriented in this direction, whereas above 800° completely new crystals are formed which grow very rapidly and are unevenly oriented. The hardness and tensile strength of the rolled sheet remain constant up to 200°, when there is a sudden fall to about half the previous values accompanied by a rapid increase in ductility. Above 700° hardness, ductility, and tensile strength all slowly decrease. The size of the grains formed by low-temperature recrystallisation is so small that they can be distinguished only at a magnification of 1500, but the röntgenogram of the recrystallised metal shows typical signs of recrystallisation having taken place. A. R. POWELL.

Electrical conductivity of certain light aluminium alloys and copper conductors as affected by atmospheric exposure. E. WILSON (J. Inst. Elect. Eng., 1925, **63**, 1108—1114).—The effect has been studied of atmospheric exposure, over a period of 24 years, on the electrical conductivity of some light aluminium alloys containing copper, nickel, manganese, and zinc in amounts not exceeding 1 or 2%. The conductivity of alloys containing copper alone or copper and manganese together diminishes continuously with time. The conductivity falls more rapidly as the copper content increases, the useful life of an alloy containing 2·61% Cu being limited to a few years. When copper and nickel are present, or copper and zinc, or combinations of all three, the conductivity at first decreases and then in some cases increases, finally reaching an approximately constant value. In two alloys containing copper and nickel there was initially a small increase in conductivity. After 7 years the conductivity of one (1·08% Cu, 1·29% Ni), dropped to about 84%, but recovered to 88·5% of its original value after 24 years. The percentage increase in electrical resistance of annealed high-conductivity copper is greater during the first year than it is for the hard-drawn variety. After exposure for 4 years the percentage increase is somewhat lower in the case of the hard-drawn wire. After a further 6 years, during which the specimens were kept loosely coiled up and not exposed, a small diminution in electrical resistance was observed. M. COOK.

Influence of the ageing temperature on the physical and chemical properties of lautal. K. L. MEISSNER (Z. Metallk., 1925, **17**, 369—373).—The effect of ageing lautal (aluminium with 4% Cu and 2% Si) for 16 and 24 hrs. at temperatures up to 200° has been investigated. The maximum hardness is obtained at 150° and the minimum ductility at 165°. The elastic limit and ultimate strength rise slowly up to 50°, remain constant between 50° and 100°, and finally reach a maximum at 165°, which is therefore the critical ageing temperature (*cf.* B., 1925, 321). Parallel with the increase in hardness and decrease in ductility the resistance to corrosion by sea-water diminishes with rise of ageing temperature, so that the alloy in most respects resembles duralumin, except that the changes that take place are less complicated and therefore more easily controlled. A. R. POWELL.

Endurance properties of non-ferrous metals. D. J. MCADAM, JUN. (Trans. Amer. Inst. Min. Met. Eng., Oct., 1925. Advance copy. 22 pp.).—Stress-cycle curves for nickel, nickel–copper alloys, and other non-ferrous metals, are similar in form to the rotating-cantilever graphs obtained for steels in previous work (*cf.* J., 1922, 60 A), and it is maintained that non-ferrous metals have endurance limits as definite as those of ferrous metals. M. COOK.

Solubility of the metals of cooking utensils, and the determination of dissolved metals. K. K. JÄRVINEN (Z. Unters. Nahr. Genussm., 1925, **50**, 221—225).—The effects of 1% hydrochloric acid, 5% sodium chloride, and 30% sugar solutions on various metals was investigated. The metal dissolved was determined by colorimetric methods, of which details are given. Pure iron was found to be more soluble than cast iron. Sugar solutions in general dissolved more metals than salt or dilute acid solutions. Iron, aluminium, zinc, nickel, and tin were more readily dissolved when pure than when alloyed. Alloys of copper, nickel, and zinc were among the most soluble and the low solubility of iron–chromium alloys was notable. A. G. POLLARD.

Leaching copper matte and speiss with nitric acid. SIXT (Chem.-Ztg., 1925, 49, 943).—When finely divided leady copper matte is treated with a slight excess of nitric acid over that required to dissolve the metals a vigorous reaction takes place and the whole of the copper dissolves leaving a residue of lead sulphate, antimonic and stannic acids, and sulphur. The solution, which contains a small amount of lead, is adjusted to an acidity of 50 g. of free nitric acid per litre and electrolysed with a magnetite anode to remove the bulk of the copper. The partially exhausted electrolyte is used for dissolving further quantities of matte after adding fresh nitric acid. If a speiss containing lead, copper, arsenic, antimony, and tin is similarly treated the solution will contain the copper and lead with most of the arsenic as arsenic acid. The lead may be removed with sulphuric acid with or without a preliminary crystallisation of lead nitrate and the copper subsequently precipitated electrolytically. Arsenic acid is removed as it accumulates in the electrolyte by the addition of lime. A. R. POWELL.

Electrolytic method for the determination of zinc in zinc ores. R. E. SULLIVAN and H. S. LUKENS (Chem. News, 1925, **131**, 321—325).—The ore is dissolved in dilute nitric acid and the solution is treated with an excess of potassium hydroxide. The precipitate is collected in a Gooch crucible, washed with hot water, dissolved in hydrochloric acid, and re-precipitated with ammonia. The combined filtrates are diluted to 350 c.c. and treated with a further quantity of potassium hydroxide to make 20 g. in all. The solution is heated to 60° and electrolysed with 8 amp. at 5—6 volts, using a platinum

anode and a revolving nickel crucible having an immersed area of about 30 sq. cm. as cathode. Complete deposition is effected in about 45 min. Without stopping the current the spent electrolyte is siphoned off while a stream of distilled water is allowed to flow into the vessel to maintain the level of the liquid constant. When current ceases to flow the cathode is removed, washed first in alcohol, then in ether, dried, and weighed. A. R. Powell.

Behaviour of lead anodes in the electrolysis of zinc sulphate solutions. H. Hock and F. Klawitter (Metall u. Erz, 1925, 22, 377; from Chem. Zentr., 1925, II., 1784).—Corrosion of lead anodes used in the electrolysis of zinc sulphate solutions is caused by peeling off of the lead peroxide, and by the presence of chlorides. The lead consumption can be reduced to about 0·5% of the weight of zinc obtained, by using solutions free from chlorides, by constructing the anode of pure material, and by protecting the apparatus from knocks and the solution from unnecessary agitation. The anode is subjected to a preliminary electrolytic treatment for several days in a dilute sulphuric acid bath, using a current density of 20—50 amp. per sq. m. L. A. Coles.

Electrolysis of lead and tin ores. R. Saxon (Chem. News, 1925, **131**, 324—325).—Electrolysis of galena in various salt solutions or in hydrochloric or nitric acid gives only a poor deposit of lead or none at all, whereas cerussite and cotunnite yield fairly good deposits of the metal when electrolysed in solutions of alkali nitrates or in dilute nitric acid. Tin may be obtained by electrolytic reduction of cassiterite in solutions of alkali sulphates or chlorides. A. R. Powell.

Steel bomb calorimeters. Kohen.—See II.

Corrosion of copper by kerosene. Staudt.—See II.

Control of electric furnaces. Schlumberger.—See XI.

Patents.

Steel alloy for the rollers of Pilger rolling mills. M. Peters (U.S.P. 1,558,918, 27.10.25. Appl., 19.3.25).—The steel contains about 2% Cr, 1·5% W, and 1·5% Ni. M. Cook.

Heat-resisting alloy steel. G. R. Rich (U.S.P. 1,560,099, 3.11.25. Appl., 27.4.25).—An alloy steel having high resistance to oxidation when hot contains 7—8% Cr, 1·25-2·25% Ni, 0·45—0·65% Mn, 0·5—1·0% Si, 0·35—0·45% C, and S and P together under 0·03%. T. H. Burnham.

Resistant-surface alloy of iron. G. H. Charls, Assr. to United Alloy Steel Corp. (U.S.P. 1,560,207, 3.11.25. Appl., 14.4.24).—The alloy contains 0·50—6·0% Si, 3·0—30·0% Cr, and not more than 0·35% Mo. M. Cook.

Treatment of ores, concentrates, and metallurgical products. H. T. Durant and P. W. Rhodes (E.P. 240,888, 8.5.24). — Metalliferous materials containing sulphides are oxidised by treatment in a mechanical agitator with a solution containing chromic acid. The solution obtained is purified if necessary and subjected to electrolysis, *e.g.*, in a diaphragm cell, to recover the metals and effect regeneration of the chromic acid for use again. T. H. Burnham.

Recovering cyanogen from solutions. L. D. Mills and T. B. Crowe (E.P. 241,669, 3.9.24).—Spent leach liquor containing cyanide is agitated in a closed vessel through which air charged with sulphur dioxide is passed. When the liquor becomes acid it is pumped into the top of a closed vessel on to a distributing plate and thence falls through a series of perforated baffle-plates in a finely divided shower while a rapid current of air is passed upwards through the vessel to remove the hydrogen cyanide from the solution. The air containing the hydrogen cyanide is passed upwards through a similar vessel through which a shower of an alkaline liquor is constantly falling, whereby the hydrogen cyanide is absorbed, with the regeneration of alkali cyanide for further leaching operations. If the original liquor contains copper, hydrochloric or sulphuric acid must be used to liberate the hydrogen cyanide and the acid liquor may be treated for the recovery of copper by known methods. A. R. Powell.

Producing and working metallic compositions containing large proportions of nickel. Western Electric Co., Ltd. From Western Electric Co., Inc. (E.P. 241,756, 24.12.24).—Nickel-iron alloys are made by melting the components, preferably in an electric induction furnace, allowing the alloy to solidify, just melting it again, and immediately casting it. The product thus obtained is readily workable without annealing. A. R. Powell.

Smelting furnace and method of smelting ores therein. (a, b) G. W. Prince and A. Douglas, Assrs. to United Verde Extension Mining Co., (c, d) J. A. Church, jun., Assr. to United Verde Extension Mining Co. (U.S.P. 1,559,508–11, 27.10.25. Appl., 9.6.25).—(a) The ore is continuously fed into the furnace chamber of a reverberatory furnace so that a continuous longitudinal mound is formed and maintained with its apex spaced from the chamber side walls. (b) The feed of the ore is regulated so that a number of continuous longitudinal mounds are formed with the apices spaced from each other and from the side walls. (c) The ore is fed in such a manner that gradually widening valleys are produced for the free flow of molten material from the firing end towards the flue end of the furnace. (d) The furnace has unfettled side walls and the ore is fed into the chamber to form two continuous longitudinal mounds of gradually decreasing height from the firing end towards the flue end of the furnace and with the apices spaced from each other and from the side walls. The portions of the side walls exposed to the molten material are made of a noncorrosive substance. M. Cook.

Treatment of metallurgical slag. H. H. Stout (U.S.P. 1,559,803, 3.11.25. Appl., 25.11.21).—A charge of molten metallurgical slag is brought into contact with an excess of non-molten material which precipitates the metal values in the slag. The materials are mixed thoroughly but the temperature is not raised during the reaction period. The excess of the added material remains in the solid state. C. A. King.

Concentration of ores. C. P. Lewis, Assr. to Minerals Separation North American Corp. (U.S.P. 1,560,170, 3.11.25. Appl., 31.3.24).—An acid pulp of the ore is agitated with a mineral frothing agent and a sulphur derivative of carbonic acid yielding anions and cations in solution, whereby a froth containing a large proportion of a mineral in the ore is produced and is then separated. T. H. Burnham.

Electrostatic separation of mineral mixtures [ores]. W. Herz (G.P. 406,669, 4.12.23).—The ore is ground sufficiently fine to liberate the various constituents and made into a pulp with water containing an electrolyte in solution. The pulp is passed over a series of conducting or semi-conducting surfaces covered with a poorly conducting skin such as that produced on the surface of a metal by oxidation. The skin may be produced during the course of the separating operation by electrostatic valve action or by the use of the Johnsen–Rabeck effect; in the latter case the semi-conducting surface is covered with a layer of fat to insulate the surface from the electrolyte. The electrolyte and conductor are connected through resistances to opposite poles of the same source of current. A. R. Powell.

Purification of [metallurgical] gases. H. A. Brassert, Assr. to C. W. Andrews (U.S.P. 1,560,202, 3.11.25. Appl., 30.11.23).—Metallurgical gases are purified by passing them through materials heated to a temperature exceeding the fusion temperature of the impurities, which are thus sintered. M. Cook.

Purification of zirconium ores. Soc. d'Etude des Agglomérés (F.P. 590,732, 13.2.24).—The mineral is pulverised at 300° and then heated with sulphuric acid (*d* 1·84) at 70° to convert the iron into anhydrous ferrous sulphate, which is insoluble in the strong acid. A. R. Powell.

Concentration of ores by froth-flotation. Minerals Separation, Ltd., Assees. of C. H. Keller and C. P. Lewis (E.P. 223,860, 6.5.24. Conv., 23.10.23).—See U.S.P. 1,554,216 and 1,560,170; B., 1925, 926 and preceding. The substance added in addition to the mineral-frothing agent is an alkali xanthate.

Electrolytic manufacture of iron. "Le Fer" Soc. Anon. (E.P. 231,179, 18.3.25. Conv., 24.3.24).—See U.S.P. 1,556,408; B., 1925, 962.

Apparatus for refining metals. H. Harris (Reissue 16,213, 17.11.25, of U.S.P. 1,465,128, 14.8.23, Appl., 12.2.25).—See E.P. 184,639; J., 1922, 821 A.

Process for producing metals and alloys. S. D. Danieli and B. M. S. Kalling, Assrs. to Aktiebolaget Ferrolegeringar (U.S.P. 1,543,321, 23.6.25. Appl., 17.12.23).—See E.P. 209,742; B., 1924, 752.

Method of making alloys. R. Walter (U.S.P. 1,560,885, 10.11.25. Appl., 29.6.20).—See E.P. 142,847; J., 1921, 738 A.

Metallic composition [alloy]. E. F. Kingsbury, Assr. to Western Electric Co., Inc. (U.S.P. 1,561,247, 10.11.25. Appl., 7.9.23).—See E.P. 224,836; B., 1925, 76.

Metal and its manufacture. Preparing boron-iron alloys. Iron–boron alloy. A. Pacz, Assr. to Gen. Electric Co. (U.S.P. 1,562,041–3, 17.11.25, Appl., [A] 26.9.18, [B] 3.5.19, [C] 7.3.22. [B, C] Renewed 7.4.25).—See E.P. 144,185; J., 1920, 548 A.

XI.—ELECTROTECHNICS.

Rapid physico-chemical methods for the control of electric furnaces. I. E. Schlumberger (Chem.-Ztg., 1925, 49, 913—915).—Owing to the rapidity with which a charge is completed in an electric furnace there is insufficient time in which to test the product by chemical analysis before discharging. If, however, the same raw materials are always used in the process a determination of the sp. gr. of the product will usually yield results from which its composition may be calculated or read from a graph with an accuracy of about 0·5%. An apparatus for the rapid determination of the sp. gr. of a powdered solid consists of a burette of 3 mm. bore graduated in 0·01 c.c. divisions and provided with a funnel and stopper at the upper end for filling. The burette is half filled with benzene and supported in an Erlenmeyer flask filled with water to maintain a constant temperature and to magnify the scale divisions. A weighed quantity of the substance, powdered to 1 mm. or finer, is dropped into the burette and the rise in the height of liquid noted, the results being plotted on a graph against varying percentage compositions of the substance. The utility of the method is illustrated with reference to ferrosilicon, ferroaluminium, and calcium carbide; ferrotungsten, however, gives unreliable results as the carbon content cannot be controlled sufficiently to obtain concordant results. A. R. Powell.

Electrolytic determination of zinc in ores. Sullivan and Lukens.—See X.

Patents.

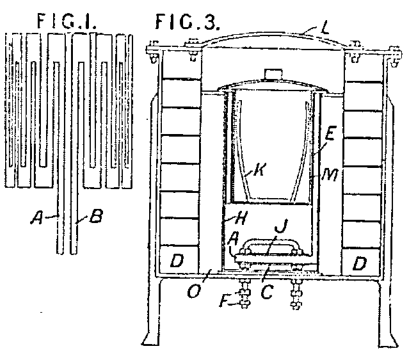
Manufacture of highly refractory bodies [filaments, *e.g.*, tungsten etc. for electric incandescence lamps etc.]. N. V. Philips' Gloeilampenfabrieken (E.P. 220,301, 31.7.24. Conv., 9.8.23).—Hafnium oxide or other compound of hafnium is mixed with the refractory metal, *e.g.*,

tungsten, and the mixture compressed to a coherent rod, which is heated in a reducing atmosphere at about 1200° and then further heated electrically at a white heat in hydrogen or other reducing atmosphere. By hammering and drawing, the rod is reduced to the desired thickness. The percentage of hafnium oxide in the finished product must lie between 0·1% and 3%. (*Cf.* Van Liempt, B., 1925, 162.) J. S. G. THOMAS.

Manufacture of coated metallic conductors [thermionic cathodes]. GEN. ELECTRIC CO., and C. J. SMITHELLS (E.P. 241,984, 28.7.24).—The conductor is coated by immersion or otherwise in a liquid consisting of a colloidal suspension of the coating material, *e.g.*, calcium oxide, reduced to a sufficiently fine state of division by treatment in a colloid mill. The coating is dried and the process repeated until a sufficiently thick, white, smooth, firmly adherent coating is produced. J. S. G. THOMAS.

Electrolytic cell. I. H. LEVIN, Assr. to GAS INDUSTRIES CO. (U.S.P. 1,560,250, 3.11.25. Appl., 4.10.21. Renewed 12.5.25).—A partitioning electrode divides a tank, containing electrolyte, closed above, and itself forming an electrode, into two communicating electrolyte compartments. The tank and electrode are of the same polarity and carry independent terminal bars. Inner electrodes of opposite polarity to the tank and partitioning electrode are arranged on opposite sides of the latter and between this electrode and the tank wall. Means are provided for preventing mixing of evolved gases at the top of the cell, and diaphragms extend from the top of the cell about each of the inner electrodes. J. S. G. THOMAS.

High-temperature resistance furnace. W. E. PRYTHERCH (E.P. 241,256, 3.4.25).*—The heating element of a high-temperature resistance furnace



consists of a cylindrical carbon or graphite crucible slotted in zig-zag fashion, as shown in Fig. 1. Connexion to the mains is made by metal U-tubes bolted to the horizontal portions of the projections, *A*, *B*, which previously formed part of the bottom of the crucible. The U-tubes pass through holes in the parts, *A*, *B*, and metal plates, *J*, *C*, on either side, and are connected to the mains by means of nuts, *F*. Water is circulated through the U-tubes. The heating element is placed between inner and outer cylinders, *E*, *H*, of non-porous refractory material, and the former contains a crucible, *K*, for holding the charge. J. S. G. THOMAS.

Electric furnace for effecting reactions between solids or liquids and gases. I. A. JULLIEN (G.P. 416,493, 6.6.23. Conv., 10.6.22).—Solids or liquids and gases are intimately mixed and charged simultaneously in the correct proportions into the upper portion of an electric furnace, by means of an automatic charging device consisting of a disc rotating with adjustable velocity, situated in a chamber provided with inlets for the powdered material and for the gas. The gas is preheated by passage through heat-exchangers situated below the reaction zone, and where the reaction to be effected is endothermic, the solid material may also be preheated by the waste heat of the gases formed during the reaction. L. A. COLES.

Electrode for discharge tubes. C. J. KAYKO, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,552,310, 1.9.25. Appl., 24.7.23).—See E.P. 219,663 ; B., 1925, 795.

Manufacture of incandescence lamps. A. DE GRAAFF and D. LELY, JUN., Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,560,981, 10.11.25. Appl., 17.11.20).—See E.P. 154,190 ; J., 1922, 245 A.

Electric conductors [copper cores covered with ferromagnetic metals applied by spraying]. O. SATTELBERG (E.P. 231,534, 31.3.25. Conv., 31.3.24).

Dehydrating oils (U.S.P. 1,559,035—6).—See II.

Removing impurities from fabrics (G.P. 416,281).—See V.

Eliminating iron from solutions of leucitic rocks (U.S.P. 1,559,179).—See VII.

Electrostatic separation of minerals (G.P. 406,669).—See X.

XII.—FATS; OILS; WAXES.

Analytical data on the oils from sharks and rays. A. ROGERS (J. Amer. Leather Chem. Assoc., 1925, **20**, 497—498).—The following data have been obtained for the oils prepared from the fresh livers rendered within a few hours after the fish had been caught. Sand shark: $d^{25\cdot5}$ 0·9216, $n^{15\cdot5}$ 1·4761, iodine value 130·4, saponif. value 186, unsaponifiable matter 0·83%. Tiger shark: $d^{25\cdot5}$ 0·9104, $n^{15\cdot5}$ 1·4711, iodine value 94·1, saponif. value 176, unsaponifiable matter 1·71%. Hammerhead shark: $d^{25\cdot5}$ 0·9218, $n^{15\cdot5}$ 1·4760, iodine value 129·7, saponif. value 184·7, unsaponifiable matter 0·62%. Nurs shark: $d^{25\cdot5}$ 0·9184, $n^{15\cdot5}$ 1·4716, iodine value 108·3, saponif. value 188·6, unsaponifiable matter 0·41%. Devil fish: $d^{25\cdot5}$ 0·9307, $n^{15\cdot5}$ 1·4840, iodine value 166·3, saponif. value 186·3, unsaponifiable matter 5·16%. Saw fish: $d^{25\cdot5}$ 0·9220, $n^{15\cdot5}$ 1·4768, iodine value 139·2, saponif. value 184·5, unsaponifiable matter 0·91%. D. WOODROFFE.

Determination of the melting point of cacao butter. T. Sabalitschka (Z. angew. Chem., 1925, **38**, 1013—1014).—The author confirms the results of several investigators according to which a sample of melted cacao butter in a capillary, even if kept in ice, requires several weeks to attain completely the solid state. Melting-point results with incompletely solidified samples are too low. Welmans' method (Pharm. Ztg., 1900, **45**, 959), in which the sample does not require to be previously melted, is coming more and more into use (*cf.* Fincke, B., 1925, 640). C. Irwin.

Usefulness of the iodine value by Margosches' method in the examination of oils. E. Stock (Farben-Ztg., 1925, **31**, 403—404).—The method of Margosches (B., 1924, 639) is strongly recommended for use in industrial practice. Results obtained for a number of oils were in close agreement with those when using Hübl's solution. It is doubtful, however, if the method is as safe for resins as Hübl's method, and apparently it is not satisfactory in the case of waxes. H. M. Langton.

Evolution of hydrogen peroxide by oils on exposure to light. G. F. A. Stutz, H. A. Nelson, and F. S. Schmutz (Ind. Eng. Chem., 1925, **17**, 1138—1141).—Seeds, oils, and resins from vegetable sources affect the sensitive photographic plate in a manner similar to the action of light, and evidence has been accumulated that this action is due to hydrogen peroxide evolved from the materials. In the case of oils the effect is greatly increased on exposure to light. Saturated fatty acids from the oils are inactive, and unsaturated fatty acids strongly active, therefore the phenomenon is associated with the drying of an oil, and probably with the entire process of oxidation of the oil film. Tests with various oils which were exposed to light at a distance of 36 cm. from a standard mercury vapour lamp showed that in general the drying oils reach a maximum and then decrease within two hours of exposure, whilst the non-drying oils show a steadier increase with no maximum, in the same period. Apparently in the case of drying oils a rapid reaction chiefly on the surface takes place with the formation of a skin relatively impervious to hydrogen peroxide. Of various metal soaps added to linseed oil, those that greatly accelerated drying showed no effect, whereas those that accelerated drying but little had a strong effect. The primary oxidation of an oil is probably a molecular autoxidation, whereby molecules of oxygen are added at the double linkings, thus forming peroxides. Further action probably results in the breaking up of these peroxides with the evolution of hydrogen peroxide as well as such products as lower acids, aldehydes, carbon dioxide, and water. A film of linseed oil continued to evolve hydrogen peroxide until it became a brittle mass. Accepting the evolution of hydrogen peroxide as a measure of the progress of the destructive oxidation, then the sources of light used in accelerated testing should have a decided maximum in the region of the spectrum 4300 to 3000A. H. M. Langton.

Fat in cacao products. Lepper and Waterman.—See XIX.

Patents.

Separation of oils or fats [from blubber and oil-bearing tissues of marine animals]. Chemical Engineering Co. (Manchester), Ltd., J. W. Spensley, and J. W. Battersby (E.P. 241,276, 16.7.24 and 24.2.25).—By feeding strips of blubber or oil-bearing tissue into the central aperture of the high-speed pinned disc mill described in E.P. 186,462 (J., 1922, 886 a), revolving at a peripheral speed of about 20,000 ft. per min., the separate fibres in the blubber are opened up in such manner as to destroy the oil-bearing cells formed by the relative crossing of the fibres. The oil can then be separated from the fibres by keeping or by means of sieves, after slight heating if necessary. Further oil can be obtained from the fibrous residue by pressing or by centrifugal treatment. The residual fibre may then be further treated for production of gelatin. A. de Waele.

Separation of fats from animal tissues. Chemical Engineering Co. (Manchester), Ltd., J. W. Spensley, and J. W. Battersby (E.P. 241,804, 16.7.24 and 24.2.25).—The process described in E.P. 241,276 (preceding) is applied to the extraction of fats solid at the ordinary temperature, *e.g.*, beef fat and mutton fat from kel fat, lard from pig leaf, etc. After the mechanical treatment the mass is warmed to a temperature somewhat above the melting point of the fat. H. M. Langton.

Extraction of oil from palm fruit and the like. T. Dickinson, F. J. Brimley, and Nigerian Products, Ltd. (E.P. 241,297, 21.7.24).—Palm fruit or the like is treated in a closed stationary vessel supplied with superheated steam and fitted with beaters or arms, whereby the pericarp is pulped and the nuts are rendered easy to crack and open. The mass is separated from the liquid by centrifuging, and conveyed to a hollow rotary dryer supplied with hot gases ; the nuts are separated from the dry fibre in a rotary separator, then cracked, and the broken nuts screened prior to the separation of the kernels from the broken shell. A. de Waele.

Digesters or sterilisers for treating palm fruit and like nuts provided with a fibrous covering containing oil or fat. C. Downs and R. A. Bellwood (E.P. 241,298, 22.7.24).—A digester having a greater height or length than its diameter is provided with a rasp-like abrasive liner on its inner surface, and with a shaft and stirrers similarly furnished with rasp-like faces on their exterior surfaces, the faces being preferably inclined. The digester is provided with steam inlets at a number of points. A. de Waele.

Plant for removal of free fatty acids and other impurities from oils, fats, and the like. Lever Bros., Ltd., R. Craig, and C. E. C. Shawfield (E.P. 242,316, 9.5.24).—The plant described is for treating fats, oils, and the like (for example the purified oil of E.P. 224,928 ; B., 1925, 45) by means of

fresh steam under low pressure. The liquid material is passed into a horizontal treatment vessel, long in comparison with its width, divided into compartments by means of baffles, so arranged that steam is admitted into the liquid in each compartment. It is constructed of an alloy (containing about 90% of aluminium together with copper, with or without a small proportion of other metal or metals, *e.g.*, nickel or magnesium), which is not corroded by free fatty acids even when hot. A substantially uniform low absolute pressure is maintained throughout the vessel, which is heated so that the free fatty acids are eliminated with the steam, and a constant level of liquid is maintained in it. Air is prevented from obtaining access to the treated oil until it is cool.
D. G. Hewer.

Increasing the viscosity of oils. E. H. Zollinger (E.P. 215,334, 15.4.24. Conv., 30.4.23).—Highly viscous oils, soluble in mineral oils, are obtained by partly oxidising fatty oils, *e.g.*, rapeseed oil, cottonseed oil, the glyceride of triricinoleic acid (*cf.* E.P. 112,624; J., 1919, 427 a), etc., and thereafter heating to 200° for several hours. In place of fatty oil a fatty acid may be used, which has already been thickened by any well-known process.
A. de Waele.

Purification of animal fats and oils. R. Vidal (F.P. 590,400, 18.11.24).—Animal fats and oils are rendered colourless and odourless, and freed from impurities by treatment with gaseous sulphur dioxide in the presence of water, with or without the application of heat and pressure. A. R. Powell.

Production of solid alcohol structure soaps. R. Falck (E.P. 242,444, 17.11.24).—Solid alcohol soaps of high melting points are produced by heating 35—40 pts. by weight of soap, desiccated to 5% of water, in 50—60 pts. of alcohol (about 96% by vol.), under a pressure of 1·5—10 atm. at 80—150°, *i.e.*, above the b.p. of alcohol. These soaps may be mixed with ordinary amorphous jelly soaps for medicinal and pharmacological purposes.
D. G. Hewer.

Manufacture of high-percentage durable soap. A. Welter (U.S.P. 1,560,626, 10.11.25. Appl., 8.1.23).—See E.P. 202,710; J., 1923, 1032 a.

Viscosimeters.—(E.P. 241,652).—See I.

Degreasing wool. (E.P. 241,314).—See V.

Treating soya beans. (E.P. 241,249).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Determination of asbestine in lithopone paint. F. G. Germuth (Ind. Eng. Chem., 1925, **17**, 1150).—1 g. of the dry sample is treated with acid ammonium acetate, filtered, the filter and its contents (asbestine plus barium sulphate) are transferred to a platinum crucible and ignited; fusion mixture is then added and the crucible heated to 600—700° for 30 min. The contents of the crucible are then treated with hot water, and a slight excess of dilute hydrochloric acid (1:1) is added to extract the barium carbonate formed. The residual asbestine is collected in a Gooch crucible and dried at 105—110° to constant weight. D. Woodroffe.

Unsaponifiable constituents of commercial rosins, with notes on rosin oil and on the polymerisation of turpentine. E. Knecht and N. B. Maurice (J. Soc. Dyers and Col., 1925, **41**, 356—361).—A high-grade pale French rosin on treatment with aqueous sodium hydroxide (twice the quantity required for neutralising the acid present) yielded 4·5% of its weight as a thick yellow oil. This consists of (1) *l*-pinene, (2) an inactive yellow oil, $C_{20}H_{32}$, b.p. 270—278° d^{20} 0·9382; (3) an oil, $C_{20}H_{32}$, b.p. 315—320°, d^{20} 0·95; both (1) and (2) are completely oxidised in acid permanganate solution and react with 8 atoms of bromine per mol. of oil; (4) an oil $C_{19}H_{30}$, b.p. 330—335° identical with colophene. Rosin Yaryan (South America) treated similarly yielded 14% of unsaponifiable material giving the same fractions and, in addition, a diterpene, $C_{20}H_{32}$, b.p. 360—370°, d^{20} 0·9572. Spanish and Portuguese rosins after the removal of abietic acid (cf. Blanes, J., 1915, 878) contained respectively 4·5% and 4% of unsaponifiable matter identical with that from French rosin. Gum Thus contains pinene and the oils (2) and (3) above, but no colophene. The temperature at which rosin distils is 320—370° and not 200° as usually stated. Rosin oil (after extraction of abietic acid with sodium hydroxide) consists of colophene and the three oils of composition $C_{20}H_{32}$ (above), and abietic acid when distilled yields the same products. Turpentine heated under pressure at 295° for 24 hrs. is changed to a thick yellowish-brown oil consisting of dipentene and the same three diterpenes obtained from rosin and rosin oil. Polymerisation of turpentine at 0° by means of 85% sulphuric acid gives a dipentene and the two diterpenes of b.p. 268—275° and 315—320°, identical with those produced by heat. It appears therefore probable that the diterpenes found in commercial rosin result from the polymerisation of turpentine under the action of heat, but from the examination of Gum Thus it is concluded that two of the diterpenes are also present in the unsaponifiable part of the resin as it comes from the tree and are produced by the cold polymerisation of turpentine. The colophene isolated from terebenthene by Deville (Annalen, 1841, **37**, 193) is not identical with that from abietic acid but is really a diterpene. The resin contained in coal does not appear to be a resin acid but is related to the higher terpenes and is probably a tetraterpene.
A. Coulthard.

Linoleum and triolin. A. Simon (Chem. Umschau, 1925, **32**, 272—275).—In triolin, the oxidised oil and resin cement of linoleum are replaced by a mixture of nitrocellulose and non-inflammable material such as tricresyl phosphate etc., this base being compounded while warm with the usual cork, fillers, etc. The tendency to slippage is somewhat less for triolin than for linoleum. The tensile

strengths of the two products are not appreciably different. Triolin is harder than linoleum, but its elastic recovery is only 60% of that of linoleum. The resistance to wear is 20% higher for linoleum than for triolin. Linoleum is not altered by treatment with steam at 130—335°, but triolin develops wart-like excrescences, and disintegrates to a soft mass with formation of yellow tar. Linoleum is also considerably more resistant to a direct flame, and whilst the fumes from burning linoleum are apparently not toxic, those from triolin killed a mouse in about 1 min., the toxicity being due to the presence of carbon monoxide, nitric oxide, and hydrocyanic acid. Even at as low a temperature as 20°, nitric anhydride appears to be evolved when air is led over triolin. A. DE WAELE.

Evolution of hydrogen peroxide by oils on exposure to light. STUTZ, NELSON, and SCHMUTZ. —See XII.

PATENTS.

Manufacture of lithopone. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER U. CO. (E.P. 241,795, 27.3.25).—Dried crude lithopone, travelling along a rotary furnace, is heated by combustion gases, practically free from dust and oxygen, passing in the opposite direction at a temperature slightly higher than that to which the lithopone is to be heated. The heated material is finally quenched in water. D. F. TWISS.

Manufacturing wax-colour binding means. J. LORENZ (E.P. 225,189, 21.10.24. Conv., 23.11.23). —A binding medium for pigments is produced by dissolving glue, gelatin, casein, or gluten in a solution of caustic soda, potash, borax, or ammonia; molten vegetable wax, paraffin, stearic acid, and a mixture of linseed oil, turpentine, and benzine are also introduced with continuous agitation. The product may be obtained as a paste or as flakes. The powdered product, if mixed with pigments, yields "oil-wax colours," which, on the addition of water, are ready for use. Dried coatings of such colours are resistant to water. D. F. TWISS.

Treatment of condensation products of carbonide [carbamide] or its derivatives and formaldehyde. F. POLLAK (E.P. 240,840, 30.9.25. Conv., 1.10.24. Not yet accepted).—Condensation products of carbamide or its derivatives with aldehydes such as formaldehyde, in either the semi-solid intermediate form or in the final hardened form, are converted into the sol state by mechanical or chemical means. For example, the products are treated in a colloid mill or similar high-speed apparatus in the presence of water or other dispersive agent; or they are submitted to the action of suitable solvents, under heat and with or without the addition of catalytic agents; as solvent, formaldehyde is especially suitable. The process is particularly useful for the conversion of powdered waste material into homogeneous masses, in which case it is not necessary that the whole should be dissolved in the sol form. The hardened condensation product when boiled with 10% hydrochloric, nitric, or sulphuric acid, yields a solution which, on cooling, deposits a bulky white precipitate, and this, after drying, can be brought into the sol state by mechanical dispersion or with the aid of a solvent such as formaldehyde. The solutions so obtained may be used for varnishes or converted into solid masses by heating. Instead of acid solutions, solutions which produce acids when heated with the condensation products may be used, *e.g.*, ammonium salts of strong acids, esters, acid chlorides, and acid salts.

Process for treating soya beans. O. JOHNSON (E.P. 241,249, 10.6.24).—Soya beans are soaked for 12 hrs. in water at a temperature not exceeding 70°, ground or crushed in a moist state, and treated with 6 pts. of water at 70° containing an alkali, *e.g.*, borax, caustic soda, or sodium bicarbonate. The milky juice is separated from the mass, *e.g.*, by centrifuging, and further centrifuged to separate the oil. The juice is then curdled with dilute acid or alum, the curd allowed to settle, and the "whey" drawn off. The curd is repeatedly washed with water at 70°, and may be bleached by repeatedly dissolving in alkali and re-precipitating with acid. It is then freed from water by centrifugal treatment, dried in a vacuum at a temperature not exceeding 60°, and ground. The product can be used in the manufacture of paints, sizes, adhesives, etc. A. DE WAELE.

Production of pure, water-white turpentine and pine oil. C. PRAETORIUS (G.P. 414,204, 23.2.22.).—The contents of the retort are cooled during dry distillation by the introduction of water or other suitable liquid through a tube with fine openings. C. T. GIMINGHAM.

Pigments (E.P. 240,852).—See IV.

Resin from pulp-mill black liquor (U.S.P. 1,560,420).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Process for impregnating materials [with rubber]. J. J. SCHILTHUIS and D. F. WILHELMI (E.P. 228,893, 2.2.25. Conv., 8.2.24).—By treating textile material, *e.g.*, cotton yarn for motor tyres, with a solution or suspension of a hydroxide or carbonate of an alkali metal, or of a hydroxide of an alkaline-earth metal, some form of soap also being added if desired, the effectiveness of subsequent impregnation by means of rubber latex, with or without sulphur, is greatly increased. Cotton so treated is rendered much more resistant to fatigue. D. F. TWISS.

Compounding rubber. C. C. LOOMIS and H. E. STUMP, Assrs. to HEVEA CORP. (U.S.P. 1,558,688, 27.10.25. Appl., 10.3.23).—Ingredients intended for rubber are prepared by encasing the particles in a thin coating of rubber before the actual mixing operation. D. F. TWISS.

Rubber compound and its process of manufacture. E. E. A. G. MEYER, Assr. to MORGAN

and Wright (U.S.P. 1,558,701, 27.10.25. Appl., 13.3.25).—The milling of unvulcanised rubber is facilitated by the addition of latex. D. F. Twiss.

Rubber vulcanisation. S. B. Molony, Assr. to R. T. Vanderbilt Co. (U.S.P. 1,558,707, 27.10.25. Appl., 20.12.19).—Rubber is vulcanised with the aid of thiuram disulphide as accelerator. D. F. Twiss.

Vulcanising rubber. G. H. Stevens (U.S.P. 1,559,196–8, 27.10.25. Appl., [A] 25.6.23 [B, C] 26.1.25).—The hot-vulcanisation of rubber is expedited by the use of [A] tetratolyltricarbodi-imide or [B] tetraxylyltricarbodi-imide or [C] by introducing into the rubber substances such that tetraphenyltricarbodi-imide is formed during the vulcanisation process. D. F. Twiss.

Composition of matter [vulcanising rubber]. H. A. Winkelmann and H. L. Trumbull, Assrs. to B. F. Goodrich Co. (U.S.P. 1,559,925, 3.11.25. Appl., 20.9.23).—Rubber or similar material is mixed with a vulcanising agent and the condensation product of an aldehyde with a substituted thiocarbamide and is then vulcanised. D. F. Twiss.

Process of making rubber compositions. G. W. Acheson (U.S.P. 1,560,132, 3.11.25. Appl., 23.6.22).—A rubber composition is prepared by introducing a dried deflocculated inorganic solid into rubber solution, then effecting precipitation by the addition of an organic liquid, such as acetone, and finally separating the rubber mixture from the liquid components. D. F. Twiss.

Production of hydrogenated caoutchouc. H. Staudinger (G.P. 415,871, 16.4.22).—By treating rubber with hydrogen and a hydrogen carrier above 200° in the presence of solvents or under increased pressure if desired, a colourless amorphous hydrocaoutchouc is obtained which is soluble in ether, benzene, or chloroform, but insoluble in alcohol or acetone. D. F. Twiss.

Production of soft vulcanised rubber and vulcanite. Technische Chemikalien Co. (G.P. 416,677, 14.4.23).—The production of soft rubber or vulcanite by vulcanisation is facilitated and improved by introducing into the mixture before vulcanisation, unsaturated, hydroxylated, sulphur-containing compounds, such as are obtainable from petroleum or tars by extraction with pure or aqueous alcohol or acetone. D. F. Twiss.

Treating rubber. O. H. Smith, Assr. to Naugatuck Chemical Co. (U.S.P. 1,562,262, 17.11.25. Appl., 15.9.24).—See E.P. 239,849; B., 1925, 1000.

XV.—LEATHER; GLUE.

Fluorescence, a method of detecting synthetic tannins in vegetable tanning extracts. O. Gerngross, N. Ban, and G. Sandor (Collegium, 1925, 565—572).—Solutions of vegetable tanning extracts (1 : 1000) were examined under ultra-violet light. Only quebracho and tizerah gave any fluorescence and this was only slight. Solutions (1 : 1000) of synthetic tannins, on the other hand, showed distinct characteristic fluorescence which was quite distinct even in strengths 1 : 100,000. The faint yellowish-green fluorescence of quebracho and tizerah extract was repressed by adding small amounts of acid or alkali. The addition of natural vegetable tanning extracts to synthetic tannins tends to diminish the amount of fluorescence, but the latter is apparent with nearly all mixtures containing 10—20% of synthetic tannin. There is no connexion between the Procter–Hirst reaction and the fluorescence test. "Tannesco," "Ordoval G," and "Kárpáti's tannin," which give a negative or only faint reaction with the aniline–hydrochloric acid reagent, exhibit extraordinarily strong blue or bluish-violet fluorescence. The test is carried out as follows:—three drops of the diluted extract (approx. 28% of tannin) are dissolved in 5 c.c. of distilled water with as little heating as possible, filtered several times until clear, and two drops of the filtrate are diluted with 10 c.c. of distilled water and examined in a dark room by the light of a six-amp. Liliput lamp (220 volts). A control test with natural vegetable tanning material should always be made for purposes of comparison. D. Woodroffe.

Destructive and preservative effect of neutral salts upon hide substance. A. W. Thomas and S. B. Foster (Ind. Eng. Chem., 1925, 17, 1162—1164).—Portions of hide powder were treated with $M/10$—$4M$ solutions of different salts of alkali metals, and samples of the liquor withdrawn at intervals to determine the nitrogen content. Toluene was added to inhibit bacterial action. The order of hydrolysis for the most concentrated solutions after 100 days was $NaBr > CaCl_2 > LiCl > NaI = MgCl_2 > KCl = NaCl > H_2O > MgSO_4 = Na_2SO_4$. The hydrolysis increased as the concentration of the solutions increased. Solutions of magnesium and sodium sulphates are better hide preservatives than sodium chloride solutions. Hide powder immersed in strong solutions of sodium sulphate is not changed and the solution remains clear, whereas in concentrated halide solutions the hide powder particles appear smaller and slimy and the solution becomes turbid. D. Woodroffe.

Patents.

Removing hair from hides or skins. H. C. Ross, H. C. Marris, and W. Walker & Sons, Ltd. (E.P. 241,666, 1.9.24).—15 lb. of sulphur are mixed with 10 gals. of boiling water, 15 lb. of burnt lime are added, the mixture is boiled for 1 hr., allowed to settle, and the clear orange-coloured liquor is run off. It should have d 1·120. 1—10 gals. of this liquor and 5—15 gals. of commercial liquor ammonia are mixed with 1000 gals. of water in the unhairing pit. The temperature may be varied from 45° to 10° according to the result required. Skins are immersed in the liquor for 6 hrs. to 3 days until the hair is loosened. D. Woodroffe.

Material for depilating and bating hides. L. Krall, and Lengrand, Krall et Cie. (F.P.

558,132, 31.10.22).—The material contains cultures of *Aspergillus oryzæ*, with or without the addition of constituents of hides, such as elastin and keratin. For example, mixtures of rice, elastin, and keratin, are inoculated with *Aspergillus oryzæ* cultivated on rice at 36° with aeration. The product, mixed with lime, is used for depilating and bating hides, and has the property of hydrolysing only the elastic constituents, the collagens not being attacked.
L. A. COLES.

Production of tanning agents. A.-G. FÜR ANILIN-FABR., Assees. of T. MARIAM (G.P. 416,277, 27.11.20).—Aluminium or chromium salts of the products obtained by the successive or simultaneous action of bisulphites, alone or together with sulphites, and aliphatic aldehydes or substances yielding them, upon aromatic nitro-compounds, such as nitrobenzene, nitrotoluene, nitronaphthalene, and nitrophenanthrene, are used as tanning agents. The products do not split off sulphuric acid during the tanning process. For example, a mixture of nitrobenzene, 10% sodium bisulphite solution, and 30% formaldehyde, is heated to 130—150° under pressure for several hours, and the product is converted into its aluminium salt by the addition of the calculated quantity of saturated aluminium sulphate solution. After separation of sodium sulphate, the solution is used for tanning, or the aluminium salt may be precipitated by the addition of alcohol, as a golden yellow syrupy product, which on drying forms an amorphous powder readily soluble in water.
L. A. COLES.

Tanning process. H. FRIEDENTHAL (G.P. 416,508, 26.7.22).—Intimate mixtures of strong solutions of vegetable or mineral tanning agents with fatty substances or hydrocarbons not miscible with the solutions, are used as tanning agents, the compositions being rubbed into the skins. For example, oak bark extract containing about 30% of tannin is worked up at 30° to a paste with vaseline of m.p. 25°, or 30% ferric chloride-potassium chloride solution is mixed with coconut oil softened by heating. L. A. COLES.

Manufacture of colloidal substances [glue] in the form of small balls or grains. A. OBERSOHN, W. WACHTEL, and P. ASKENASY (U.S.P. 1,559,126, 27.10.25. Appl., 7.1.25).—A hot concentrated solution of glue is sprayed under pressure to form drops, which are passed into and through a gaseous cooling agent to which pressure is applied to retard the rate of passage of the drops through it. D. WOODROFFE.

Dyeing leather (G.P. 417,209).—See VI.

XVI.—AGRICULTURE.

Two unusual colloidal soils. C. F. SHAW (Soil Sci., 1925, **20**, 419—423).—A soil material from the dried bed of a lake in California contained from 134 to 540% of water in the field condition without drainage. The moisture equivalent was from 348 to 380, and the loss on ignition 38 to 54%. The soil had a horny texture when dry and consisted of unweathered material, the inorganic matter being mainly silica. A subsoil from the island of Hawaii, of an elastic and cheese-like consistency, and formed by weathering of lavas under high rainfall and temperature, contained about 375% of water.
C. T. GIMINGHAM.

Critical p_H for the formation of hardpan in acid clay soils. J. R. SKEEN (Soil Sci., 1925, **20**, 307—311).—The p_H value in the neighbourhood of natural hardpans occurring in an acid, heavy clay soil ranged only from 4·7 to 4·95. The hardpan was cemented by iron and aluminium compounds for the most part, and experiments with artificial soils showed that ferric hydroxide may be precipitated on kaolin, giving a hardpan analogous to that found in the field at p_H values lying between 4·4 and 5·0. It is concluded that there is a critical p_H for hardpan formation in an acid clay soil. C. T. GIMINGHAM.

Determination and control of the potassium and phosphorus content of the soil in practice. E. MÖLLER-ARNOLD (Z. Pflanz. Düng., 1925, **B4**, 503—511).—The various methods which have been suggested for determination of the manurial requirement of soils as regards potassium and phosphoric acid are considered from the point of view of their direct economic value to the practical agriculturist. The methods discussed include both laboratory tests and field trials. C. T. GIMINGHAM.

Alkali [soil] studies. III. Tolerance of barley for alkali [salts] in Idaho soil. R. E. NEIDIG and H. P. MAGNUSON (Soil Sci., 1925, **20**, 367—391 ; *cf.* B., 1925, 182, 292).—The results of pot experiments are discussed in which 4 successive crops of barley were grown in soil treated with varied amounts of sodium chloride, carbonate, and sulphate singly and in combination. In general, with all treatments, the salts were most toxic to the first crop, stimulated the second crop, and were somewhat toxic to the third crop. With the fourth crop, the yields tended to approach the normal. Mixtures of all three salts, in most cases, increased the yields of all crops except the first. Different soils behave very differently towards added alkali salts, and the tolerance of crops for these salts varies widely in different soils.
C. T. GIMINGHAM.

Disintegration of limestone and dolomite separates as influenced by zone of incorporation. W. H. MACINTIRE and W. M. SHAW (Soil Sci., 1925, **20**, 403—417).—Limestone and dolomite, ground to varying degrees of fineness, were incorporated with soil in lysimeters under field conditions. After 4 years, the extent of decomposition was measured by determination of the residual carbonate. The soil used had never been limed and had a p_H value of 6·23. With both limestone and dolomite, the most finely-ground material ("80—200-mesh") was practically completely decomposed, whether mixed with the surface soil or lower layer of soil. The coarsest material ("10—20-mesh") showed little change;

other grades were intermediate. Limestone, in each case, showed greater decomposition than the corresponding grade of dolomite. Both materials, of each degree of fineness, were more completely decomposed in the lower soil layer than in the surface layer.
C. T. GIMINGHAM.

Nitrate accumulation under a straw mulch. W. A. ALBRECHT and R. E. UHLAND (Soil Sci., 1925, **20**, 253—267).—Experiments in pots and in the field on the causes of the reduced accumulation of nitrates under a heavy straw mulch (*cf.* Albrecht, J., 1923, 153 A) indicate that, by cutting down evaporation and aeration and thus increasing the moisture content and lowering the temperature, the mulch induces a physical condition of the soil unsuitable for nitrification. Ammonia is present in larger quantities under a mulch than in soil not mulched.
C. T. GIMINGHAM.

Residual effects of 40 years' continuous manurial treatments. II. Effect of quicklime on soil treated with dung. J. W. WHITE and F. J. HOLBEN (Soil Sci., 1925, **20**, 313—327; *cf.* B., 1924, 990).—A study of the condition and amount of soil organic matter on two series of field plots after 40 years of continuous treatment and cropping; one series having been manured with dung every two years at the rate of six tons per acre and with quicklime every four years at two tons per acre, and the other with dung only. Determinations were made of total nitrogen, organic carbon, and of organic matter and nitrogen soluble in 3% alkali. In general, the results indicate that the soil receiving both dung and lime has utilised the organic matter more completely, has produced a higher yield of dry matter, and, at the end of the period, contains more organic matter and nitrogen than the soil receiving dung only. From a consideration of the relation found to exist between the residual organic matter and the total yields of dry matter on similarly treated plots throughout the series, it is estimated that 93% of the organic matter on the lime and dung plots and 88% on the plots treated with dung alone is derived from crop residues.
C. T. GIMINGHAM.

Action of copper compounds on "stinking smut" of wheat. BODNAR and TERENYI.—See XIX.

Clays as emulsifiers for mineral oils. YOTHERS and WINSTON.—See XXIII.

Spreaders for spray materials. ROBINSON.—See XXIII.

Mercuriferous germicides for pickling grain. KRAUSS.—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

PATENT.

Extracting sugar from beets. I. DE VECCHIS, Assr. to HOME AND COLONIAL INVESTMENTS, LTD. (U.S.P. 1,562,151, 17.11.25. Appl., 5.12.23).—See E.P. 211,113; B., 1924, 991.

XVIII.—FERMENTATION INDUSTRIES.

Determination of diastase in malt extract. SEELIGMANN (Chem.-Ztg., 1925, **49**, 943).—The malt extract is mixed with water to give a 4% solution, which is filtered, and 10 c.c. of the filtrate are digested at 40° for exactly half-an-hour with 250 c.c. of a 3% solution of arrowroot starch; 3 c.c. of 10% sodium hydroxide solution are added, and the mixture is cooled rapidly and diluted to 300 c.c. The solution is transferred to a burette and used for the titration of 25 c.c. of a dilute Fehling's solution standardised as usual with pure maltose. The maltose content of the original extract is determined by titration against the same Fehling's solution, and the increase in maltose in mg. per g. of extract after treatment with the starch gives the diastatic power of the malt in Pollak units.
A. R. POWELL.

Volutin in yeast cells. M. GLAUBITZ (Z. Spiritusind., 1925, **48**, 363).—Volutin, a nucleic acid compound, is stained blue when yeast, killed in 40% formalin, is treated first with methylene-blue (0·4% in 25% alcohol) and then with 1% sulphuric acid. It appears as globules or flakes, and may occupy up to 90% of the cell volume. The volutin content of yeast, produced by the aeration process, increased during the first 4 hrs. and then decreased to one-third by the eighth hour. In a series of 30 experiments no connexion could be detected between volutin content and fermenting power of the yeast, nor between volutin and the nitrogen content. Pressed yeast, kept at room temperature, suffered no diminution of volutin in 6 days, but after 12 days the cells were free from volutin.
G. T. PEARD.

Direct and indirect methods for the determination of dry matter, and the determination of sugar in vinegar. G. REIF (Z. Unters. Nahr. Genussm., 1925, **50**, 181—192).—Comparative experiments show that direct and indirect methods do not give similar figures for determinations of dry matter in synthetic vinegars. For this determination in fermentation vinegars the indirect method of Lehmann and Gerum (Z. Unters. Nahr. Genussm., 1912, **23**, 267; *cf.* Fresenius and Grünhut, J., 1920, 555 A) is advocated. For the determination of sugar in vinegars the iodometric method, coupled with the use of the sugar tables of Auerbach and Bodländer (cf. J., 1922, 991 A), is shown to be very satisfactory.
A. G. POLLARD.

Determination of tannic acid in fermentation vinegar. G. REIF (Z. Unters. Nahr. Genussm., 1925, **50**, 192—195).—The method described is as follows:—A reagent is prepared by dissolving 3 g. of sodium tungstate, 2 g. of sodium phosphate, and 0·05 g. of molybdic acid in 25 g. of warm water, heating the solution on a water bath, and adding concentrated nitric acid, drop by drop, until the solution is neutral to litmus. 10 c.c. of the vinegar are treated with 0·5 c.c. of 10% hydrochloric acid and 1 c.c. of the reagent, heated quickly to boiling and maintained at that temperature for 2 hrs. The violet coloration produced is compared with that of a standard solution. Tannic acid can be determined by

this method in dilutions of 1 : 500,000. Tannic acid is absent from synthetic vinegars, and with mixtures of synthetic vinegar and fermentation vinegar the coloration produced is proportional to the amount of the latter present. A. G. POLLARD.

PATENT.

Production of hop extracts. ELEKTRO-OSMOSE A.-G. (GRAF SCHWERIN GES.), Assees. of P. KOLBACH, W. WINDISCH, and W. DIETRICH (G.P. 413,913, 10.12.22).—Free acids present in hops, hop extracts, waste material from hops, and by-products from brewing processes, are neutralised by boiling with alkaline solutions in which the maximum concentration of hydroxyl ions is 0·005 g.-mol. per litre. The extracts, which are concentrated in the usual manner, contain the bitter constituents in a soluble form. L. A. COLES.

XIX.—FOODS.

Action of copper compounds on "stinking smut" of wheat. J. BODNÁR and A. TERÉNYI (Chem.-Ztg., 1925, **49**, 902).—When treated with salts of copper, spores of "smut" absorb copper, the amount taken up depending on the compound used. Inhibition of germination of the spores occurs if they absorb 0·4% or more of their weight of copper. It is, therefore, important that copper compounds used in the dry state for combating this fungus, should be sufficiently soluble in the soil water to keep the concentration of copper above that necessary to prevent germination. C. T. GIMINGHAM.

Rational milling of maize. D. MAROTTA and F. DI STEFANO (Annali Chim. Appl., 1925, **15**, 227—238).—Roll mills give much better results than the ordinary stones when applied to maize, since they allow of the removal of the germ and of the better separation of the bran constituents. T. H. POPE.

Determination of sucrose in condensed milk. K. SCHERINGA (Pharm. Weekblad, 1925, **62**, 1034—1035).—In a 200 mm. tube at 20° the rotations of an inverted 10% solution of anhydrous lactose and an inverted 10% sucrose solution are respectively + 11·1° and −4·0°. In view of the fact that the cupric-reducing power of dextrose is very nearly equal to that of inverted anhydrous lactose, the proportion of sucrose in condensed milk may be determined as follows: 33·33 g. of the milk are dissolved to 100 c.c. in the usual way and the bright, filtered solution is inverted. If this inverted solution has the rotation b° and a reducing power corresponding with a% of dextrose, the percentage of sucrose in the total sugars is given very nearly by $(11-10b/a)/0{\cdot}151$. A slightly more accurate result is obtained by substituting $0{\cdot}993a$ for a. T. H. POPE.

Chemistry of the ripening of cheese. II. W. GRIMMER and B. WAGENFÜHR (Milchw. Forsch., 1925, **2**, 193—198; Chem. Zentr., 1925, **96**, II., 1718).—The percentages of nitrogen and sulphur in "caseoglutin," obtained from several different kinds of cheese, differ from those given by earlier workers. "Caseoglutin" is considered to be a group of substances, differing in elementary composition, but having the same origin in cheese. Details as to its solubility and specific rotation in various solvents are given. Three times as much tryptophane can be obtained from it as from casein, but it does not contain three times as much sulphur; it is regarded as a mixture of the sulphur-free and sulphur-containing components of casein. C. T. GIMINGHAM.

Determination of fat in cacao products. H. A. LEPPER and H. C. WATERMAN (J. Assoc. Off. Agric. Chem., 1925, **8**, 705—710).—The prepared sample (2—3 g.) is weighed into a Knorr extraction tube containing a tightly packed mat of purified asbestos (washed with alcohol, ether, and petroleum benzine) and the tube inserted into the rubber stopper of a filtering bell-jar which is connected to suction through a two-way stopcock. A 150-c.c. flask of known weight is so placed that the stem of the tube passes through its neck. The tube is filled two-thirds full of petroleum benzine (re-distilled below 60°), the sample stirred with a flat-ended glass rod, left to stand for 1 min., and the tube drained by suction. More solvent is added while the tube is rotated and the sides are washed down. About 10 extractions are necessary for complete removal of the fat. The tube is then disconnected, traces of fat are washed from the stem, the solvent is evaporated, and the contents of the flask are dried to constant weight. The difference between the figures given by this method and the higher ones by that of the Assoc. of Off. Agric. Chemists, which uses anhydrous ether as solvent, is shown not to be due to fat but chiefly to theobromine. The fat extracted by petroleum benzine is free from sugar and cacao alkaloids. D. G. HEWER.

Solubility of metals of cooking utensils. JÄRVINEN.—See X.

PATENTS.

Desiccation, particularly of substances of organic origin. H. L. P. TIVAL and F. A. DESCOMBES (E.P. 222,154, 22.9.24. Conv., 22.9.23).—The solution or substance to be desiccated is frozen, ground, the ground particles are introduced as a mist into an air-tight receptacle of high vacuum and low temperature, and precipitated by mechanical and/or electrical means on an endless transporting surface. Escaping vapours are precipitated or condensed, and the material during its passage through the apparatus may be more or less completely sterilised by exposure to bactericidal rays, such as X-rays, Goldstein canal rays, or those from ultraviolet ray tubes. Rotating brushes are used for stirring and projection of the mist or powder to the transporting surfaces. D. G. HEWER.

Processes of making butter. A. E. WHITE. From MILK OIL CORP. (E.P. 242,363, 12.8.24).—Butter is made without churning from pure milk oil by adding an emulsifying agent obtained from milk, such as casein or dried skimmed milk, together with water, so that the fat percentage of the mixture

is between 50 and 80. The mixture is heated above the melting point of milk oil and stirred and mixed until the size of the fat globules is diminished to approximately that found in natural milk or cream (5μ). An emulsion may be produced by means of a mechanical emulsor, or the mixture cooled to 27° and stirred until the viscosity is increased to a point where the internal friction causes emulsification. The emulsion is cooled to 18° or lower, and practically complete sudden agglomeration brought about, with expulsion of some buttermilk, by simple pressure with a wooden paddle etc., instead of by violent agitation resulting in churning as described in U.S.P. 1,509,086/8 (B., 1924, 1027). The resulting butter may be worked and treated as usual. D. G. Hewer.

Treating flour. F. L. Dunlap, Assr. to Industrial Appliance Co. (U.S.P. 1,560,045, 3.11.25. Appl., 25.7.23).—A shortening effect is produced by increasing the acidity of the flour beyond the optimum for bread making. D. G. Hewer.

Prevention of decay of citrous fruits. H. R. Fulton and J. J. Bowman (U.S.P. 1,560,558, 10.11.25. Appl., 11.9.25).—The fruits are treated with an aqueous solution of lithium carbonate at a temperature below 49°. D. G. Hewer.

Preservation of fresh fruits and vegetables. H. R. Fulton and J. J. Bowman (U.S.P. 1,560,559, 10.11.25. Appl., 14.9.25).—An aqueous solution which contains the sodium oxide and boric acid radicals in the proportion of 1 to 3, and of such strength that it will retard the development of stem end rot, blue mould rot, etc. (*e.g.*, 5 pts. by weight of borax and 0·43 of sulphuric acid in 100 of water) is applied to the surface of the fresh fruit or vegetable. D. G. Hewer.

Direct cooling [refrigeration] of goods [foods]. A. J. A. Otteson (U.S.P. 1,562,360, 17.11.25. Appl., 1.3.24).—See E.P. 211,032; B., 1924, 310.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of morphine in opium. E. Machiguchi and S. Shirono (J. Pharm. Soc. Japan, 1925, [524], 849—861).—8 g. of opium powdered and dried at 60° are shaken with 2 g. of calcium hydroxide and 80 c.c. of water for 1 hr. and filtered through a paper of 15 cm. diam. 50 c.c. of the filtrate are poured into a thick-walled bottle of 100 c.c. capacity containing 20 c.c. of a mixture of ether and benzene (4 : 1) and 1 g. of ammonium chloride, and shaken gently for 10 min. After keeping for 20 hrs. the separated crystals are collected on a filter paper of 9 cm. diam., washed four times with 5 c.c. of water, and dried below 60°. After cooling, and washing with 20 c.c. of ether–benzene (1 : 1) the alkaloid is dried first at a low temperature then at 96—100°. It is dissolved in 10*N*-hydrochloric acid and titrated with 10*N*-alkali using 3 drops of methyl red as indicator. The amount of the acid used multiplied by 0·5704 gives the percentage of morphine. The result obtained lies between those given by the official British and American methods. K. Kashima.

Bio-assay of thyroid. R. Hunt (U.S. Pub. Health Rep., 1925, **40**, 1461—1466).—The therapeutic value of thyroid cannot be ascertained by determination of the iodine content, as analysis does not differentiate between added iodine and iodine in thyroid combination. The acetonitrile method (the increased resistance to acetonitrile of white mice to which thyroid has been administered) is recommended for assay purposes. R. E. Thompson.

Examination of neoarsphenamine [neosalvarsan] and sulpharsphenamine. E. Elvove (U.S. Pub. Health Rep., 1925, **40**, 1235—1248; *cf.* B., 1924, 767).—Nearly all the oxidisable sulphur of neosalvarsan is oxidised by iodine in alkaline solution, whilst less than half of the total sulphur in sulpharsphenamine is oxidised by this procedure. This appears to be due to differentiation by iodine between organically combined sulphur and that present as uncombined sodium formaldehyde bisulphite, and not to a slow rate of oxidation of the organically combined sulphur. Advantage is taken of this difference in behaviour for the purpose of differentiating between neosalvarsan and sulpharsphenamine, and, in conjunction with other determinations, for evaluating, at least approximately, the composition of mixtures of them. R. E. Thompson.

Gas washing. Absorption with mixed absorbents. Weissenberger, Schuster, and Zack. —See I.

Ethyleneglycol as anti-freeze material. Curme, jun., and Young.—See I.

Patents.

Manufacture of hydrocyclic ω-aminoalkyl compounds. H. Rupe (E.P. 240,814, 14.9.25. Conv., 1.10.24. Not yet accepted).—Hydrocyclic ω-aminoalkyl compounds are prepared by catalytically reducing hydrocyclic cyano-compounds, and if necessary hydrolysing the intermediate products. The process is particularly suitable for the treatment of α-cyano derivatives of hydroaromatic ketones, such as camphor, menthone, thujone, pulegone, and *cyclo*hexanone. The products are basic in character and can be converted into water-soluble salts which possess therapeutic properties. For example, a solution of α-cyanocamphor is treated with hydrogen in the presence of a nickel catalyst. After the calculated quantity of hydrogen has been absorbed ($3\ H_2$ per 2 mols. of cyanocamphor), the product is filtered, the solvent distilled away, and the hydrochloride of methylenecamphor-camphomethylamine precipitated. The latter is then hydrolysed, the oxymethylenecamphor formed as by-product removed with ether, and the aqueous solution evaporated to obtain the hydrochloride of α-camphomethylamine.

Production of urea [carbamide]. L. Casale (E.P. 241,123, 19.5.25. Conv., 23.12.24).—In the

continuous production of carbamide from ammonia and carbon dioxide, economy of heat and power is obtained by utilising the gaseous mixture obtained from a catalytic synthetic ammonia plant, while still under the influence of pressure and heat.

C. O. HARVEY.

Making *iso*bornyl esters. J. EBERT, Assr. to J. C. DEHLS and L. STEIN (U.S.P. 1,555,947, 6.10.25. Appl., 13.5.20. Renewed 19.2.25).—40·8 pts. of pinene saturated with hydrogen chloride are heated under reflux for 20 hrs. with 130 pts. of glacial acetic acid to which 20 pts. of finely-divided tin or zinc and 2 pts. of finely-divided copper or iron have been added. The excess of acetic acid is distilled off under reduced pressure and the residue poured into water, whereupon *iso*bornyl acetate separates in 90% yield.

T. S. WHEELER.

Purification of butyric aldehyde. C. BOGIN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,556,067, 6.10.25. Appl., 12.5.24).—6% of water is added to butaldehyde and the constant-boiling mixture obtained is distilled at 68°, no polymerisation occurring. On cooling, 98% of the water separates, and the remainder is removed by salting out.

T. S. WHEELER.

Sterilising proteins or other colloidal material. C. A. MILLS (U.S.P. 1,556,120, 6.10.25. Appl., 25.7.22).—A serum protein or other sensitive protein, such as globulin, is dissolved in a dilute solution of sodium chloride and treated with mercuric chloride. A precipitate is produced, and sterilisation is effected. When this is complete, sodium chloride or other salt of a light metal is added, whereupon the protein is liberated from the mercury and goes back into solution, whilst the mercury salt forms a soluble double compound with the light metal salt. The inorganic salts are then removed by dialysis.

T. S. WHEELER.

Preparation of 2-nitro-3 : 4-dimethoxy-1-methylbenzene [2-nitrohomoveratrole]. E. MERCK, CHEM. FABR., Assees. of M. OBERLIN (G.P. 415,315, 21.3.24).—The compound is prepared by treating 4-acetoxy-3-methoxy-1-methylbenzene (acetylcreosole) with fuming nitric acid, hydrolysing and subsequently methylating the nitration product, *i.e.*, a mixture of 2 : 5- and 2 : 6-dinitro-4-acetoxy-3-methoxy-1-methylbenzene, so as to produce a mixture of 2 : 5- and 2 - 6-dinitro-3 : 4-dimethoxy-1-methylbenzene, and subsequently reducing the 5- and 6-nitro-groups to amino-groups, and then replacing them by hydrogen. The product is a yellow oil, b.p. 115—117° at 1 mm., and volatile in steam ; on oxidation with potassium permanganate it yields 2-nitrohomoveratric acid, m.p. 200—202°.

L. A. COLES.

Production of tartaric acid from saccharic acid. DIAMALT-A.-G. (G.P. 415,685, 4.6.18).—Tartaric acid is obtained by oxidising saccharic acid with hydrogen peroxide. For example, barium peroxide is added at 15° to a solution of saccharic acid in 5% sulphuric acid, obtained by oxidising carbohydrates with nitric acid or oxides of nitrogen in the presence of sulphuric acid, until the whole of the sulphuric acid is converted into barium sulphate. The solution is warmed gradually to 40—50°, filtered, and tartaric acid is precipitated in the usual manner, a 60% yield, calculated on the saccharic acid, being obtained. Alternatively, 30% hydrogen peroxide may be used instead of barium peroxide, the sulphuric acid being subsequently precipitated by the addition of lead or calcium salts.

L. A. COLES.

Production of a water-soluble complex bismuth compound of 7-iodo-8-hydroxyquinoline-5-sulphonic acid. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (G.P. 416,329, 18.12.23).—The compound is obtained by treating aqueous solutions of alkali 7-iodo-8-hydroxyquinoline-5-sulphonates with mannitol-bismuth nitrate or sugar-bismuth nitrate solutions, and subsequently separating the complex bismuth compound by the addition of organic precipitants, such as alcohol or acetone. The product, which contains about 13—16% Bi, is of therapeutic value.

L. A. COLES.

Conversion of methyl sulphide into carbon tetrachloride and other chlorinated compounds. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of A. HALLSTEIN (G.P. 416,603, 1.2.24).—Methyl sulphide, obtained as a by-product in the manufacture of sulphate-cellulose, is freed from all traces of mercaptans, and chlorinated vigorously in the absence of moisture, and preferably in the presence of iodine or other catalysts. For example, dry chlorine is passed at 15° into methyl sulphide free from mercaptans, until no more is absorbed. The product separates into two layers ; the upper layer contains trimethylsulphonium chloride, which is worked up into methyl chloride and methyl sulphide, and the lower layer consists of a mixture of sulphur dichloride, carbon tetrachloride, perchloromethyl mercaptan, and *methyltrichloromethylsulphur chloride*, a colourless liquid, *d* 1·642, b.p. 194° at 750 mm., which can be separated by distillation. Perchloromethyl mercaptan and methyltrichloromethylsulphur chloride are coverted into sulphur chloride and carbon tetrachloride on further chlorination. The chlorination proceeds more rapdily in the presence of iodine, and the yields of carbon tetrachloride and sulphur chloride are increased.

L. A. COLES.

Benzoxazolone-5-arsonic acid. L. BENDA, Assr. to L. CASSELLA u. Co. (U.S.P. 1,543,544, 23.6.25. Appl., 17.4.24).—See E.P. 214,628, B., 1925, 149.

Anaesthetic. H. WIELAND, Assr. to C. H. BOEHRINGER SOHN (U.S.P. 1,561,130, 10.11.25. Appl., 27.1.25).—See E.P. 205,240 ; J., 1923, 1199 A.

Manufacture of organic compounds by reactions involving replacement of substituent atoms or groups with liquid reagents. W. V. SHANNAN, Assr. to GAS LIGHT AND COKE Co. (U.S.P. 1,561,491, 17.11.25. Appl., 6.11.23).—See E.P. 212,970 ; B., 1924, 462.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Influence of photographic fixing baths and their constituents on different metals. M. Eule (Phot. Ind., 1925, 1244—1245, 1273—1274).—The action of (*a*) 20% thiosulphate solution, (*b*) sulphurous acid–sodium sulphite solution, (*c*) an acid fixing bath of (*a*) containing (*b*), and (*d*) a 20% thiosulphate bath containing silver, on lead, copper, tin, iron, aluminium, zinc, brass, and nickel-plated brass, was studied. Data are tabulated, showing the nature of any coating on the metal surface and any precipitate formed, and whether any of the metal is found in the solution, and a table is given from which a quantitative indication of the suitability of any metal for use in a given bath can be ascertained.
W. Clark.

Patents.

Utilisation of luminescent and catalytic substances [*e.g.*, for *X*-ray screens].—P. L. G. Marcotte (U.S.P. 1,546,499, 21.7.25. Appl., 31.7.22). —The fluorescence of substances under the action of *X*-rays varies according to the size of the crystals, and it is advantageous to use crystals between fixed limits of size. For example, the tungstates of zinc, calcium, and magnesium as ordinarily prepared are not efficient substitutes for barium cyanoplatinate, but when used in the form of crystals between 200-mesh and 80-mesh size they are equal and between 150-mesh and 120-mesh size are superior to the cyanoplatinate. T. S. Wheeler.

Manufacture of photographic films. Kodak, Ltd., Assees. of J. H. Haste (E.P. 232,232, 8.4.25. Conv., 14.4.24).—See U.S.P. 1,532,819; B., 1925, 479.

Colour photography. W. V. D. Kelley and D. Tronolone, Assrs. to Kelley Color Laboratory, Inc. (U.S.P. 1,561,168, 10.11.25. Appl., 5.2.24).—See E.P. 228,887; B., 1925, 866.

XXIII.—SANITATION; WATER PURIFICATION.

Water filter design. J. W. Armstrong (Eng. and Contracting, Water Works Issue, 1925, **64**, 847—856; J. New England Water Works Assoc., 1925, **39**, 254—271).—In order to obtain highest efficiency in coagulation the water should be violently agitated immediately after addition of coagulant. Later agitation is ineffective. Tests on mixing basins of the around-the-end type, having thirteen 180° turns, showed that there is a progressive increase in the rate of settling, 7—8 hrs. being required for subsidence at the first bend, while less than 2 hrs. was required at the thirteenth turn. The application of lime can be very closely controlled by a potentiometric method by measurement of the hydrogen-ion concentration. If the filters are equipped with sight glasses in which the suspended matter in the effluent is revealed by a beam of light, operation of the filters can be controlled by clarity of the effluent instead of by loss of head. Seepage of water through concrete, particularly where this is subject to frost action, leads ultimately to disintegration. Water of p_H value less than 7·5 and alkalinity less than 100 p.p.m. will dissolve calcium carbonate from concrete, and aluminium compounds also slowly go into solution under these conditions, resulting in loss of strength, the rapidity of disintegration depending largely on the porosity of the concrete. R. E. Thompson.

Fineness modulus for [water] filter sand. R. G. Tyler (Eng. and Contracting Water Works Issue, 1925, **64**, 827—832; J. New England Water Works Assoc., 1925, **39**, 239—253).—Little or no correlation could be observed between the fineness modulus of a filter sand (*cf.* Abrams, Bull. 1, Structural Materials Res. Lab., 1918; also J. Amer. Water Works Assoc., **11**, 677) and observed values of loss of head or transmission capacity in an experimental sand bed. Calculation of the surface modulus of the sand grains from the results of the sieve analysis (*cf.* Abrams and Talbot, Proc. Amer. Soc. Testing Materials, 1919, **19**) gave a better correlation, the average deviation of the calculated transmission capacity from the observed values being 24·3%, as compared with 32·6% by Hazen's method (Rep. Mass. State Bd. of Health, 1892, 550). The method of King and Slichter (19th Annual Rep. U.S. Geolog. Survey, Pt. II.), consisting of measuring the time required to pass a known volume of air through air-dried sand under specified conditions, gave an average variation from observed values of only 7·19%, but special apparatus is required for this method.
R. E. Thompson.

Properties of iron compounds and their relation to water clarification. L. B. Miller (U.S. Pub. Health Rep., 1925, 40, 1413—1419).—Experiments on precipitation of iron compounds indicate that "ferric floc" possesses many of the properties of "alum floc," an essential difference being that the aluminium precipitate dissolves readily at higher p_H values, forming soluble aluminates, while the ferric precipitate does not. A study of the composition of the precipitate from ferrous sulphate solutions showed that sulphate is at first carried down to an appreciable extent, decreasing finally to nil as increasing quantities of alkali are added. As in the case of aluminium the p_H range over which flocculation occurs is determined by the nature and concentration of the anions present. Titration curves of ferrous sulphate, ferric chloride, and ferric alum by alkali are given. R. E. Thompson.

Clarification of coloured waters. L. B. Miller (U.S. Pub. Health Rep., 1925, **40**, 1472—1481).—Experiments with specially prepared "synthetic humus" and with several "colour" samples of the humic acid type showed coagulation of the colour by electrolytes to be due to the cation, the coagulating power increasing with the valency. Addition of hydroxyl ions stabilises the colloid, rendering clarification more difficult. In treatment of water

containing humic substances with alum, clarification is effected by formation of "colour floc" precipitated by the aluminium ion, and formation of "alum floc," by removing aluminium ions from solution, is antagonistic. Therefore a p_H value below 5·4, at which point precipitation of aluminium first approaches completion, is most favourable.

R. E. THOMPSON.

Colloidal clays as emulsifiers for mineral oils used in spraying citrus groves. W. W. YOTHERS and J. R. WINSTON (J. Agric. Res., 1925, **31**, 59—65).—In the preparation of mineral oil sprays for citrus trees, soap solutions were successfully replaced by suspensions of kaolin, fuller's earth, and brick earth clay. The most satisfactory suspensions were prepared by covering the dry clay with the requisite amount of water and allowing to stand unstirred for 30 min. After stirring, the oil was added, and the whole emulsified by pumping twice through an ordinary bucket pump. The emulsion formed a thin paste which remained unchanged for an indefinite period. It covered well, and was as efficient as, and no more injurious to foliage than, soap emulsion. The clay emulsions had the additional advantage that they could be mixed with hard water and other spray materials.

A. G. POLLARD.

Spreaders for spray materials and the relation of surface tension of solutions to their spreading qualities. R. H. ROBINSON (J. Agric. Res., 1925, **31**, 71—81).—Attempts are described to correlate the efficiency of "spreader" solutions with their physical properties. Although, in general, solutions having a low surface tension or low interfacial tension to oil proved the best spreaders, no definite relationship was apparent. Other factors, notably the nature of the sprayed surface, are involved. Water-soluble proteins proved the best spreaders at low concentrations, skimmed milk neutralised with slaked lime being the most practicable preparation. Spreader solutions aided the even distribution of material over the sprayed surface, but did not increase the total amount adhering to the surface.

A. G. POLLARD.

Determination of mercuriferous germicides in liquor used for pickling grain. J. KRAUSS (Z. angew. Chem., 1925, **38**, 1088—1091).—Tests have been made to determine the concentration of the solution of germicide to be added to bring the concentration of a used solution up to the desired strength for use again. With an immersion of 30 min. and using 2 litres of a 0·25% solution of the reagent per kg. of grain, the bulk of the solution after use must be made up with a 0·65% solution in the case of "uspulun," a 0·57% solution in the case of "germisan" (17·5% Hg), and a 0·52% solution in the case of "urania" (5·8% Hg) in order to obtain the original concentration. After pickling for 5 mins. the amount of mercury adsorbed by the grain is 83% with "uspulun," 79% with "germisan," and 55% with "urania" of that adsorbed in 30 mins., which is 81, 90, and 91% respectively of that adsorbed in 1 hr.

A. R. POWELL.

Diffusion of gaseous [fumigating] mixtures through building materials. THILENIUS and POHL (Z. angew. Chem., 1925, **38**, 1064—1067).—The diffusion of mixtures of hydrogen cyanide gas with irritants, such as ethyl chloroformate or chloropicrin through ordinary building materials such as brickwork with and without plaster or wallpaper, breeze concrete with and without plaster, ordinary plaster, etc. was measured. The composition of the gas mixture was altered in passing through the material, the proportion of irritant generally decreasing. This is largely due to the influence of moisture and uncombined lime; the separation of the mixture is more pronounced with building materials containing a large proportion of fine pores. Paint, paper, etc. have very little influence on the gas mixture. The results of these experiments have an important bearing on the use of an admixture of these irritants to act as a warning in rooms adjacent to those in which hydrogen cyanide is used for fumigation purposes.

B. W. CLARKE.

Destruction of cockroaches and devitalisation of their eggs by cyanogen chloride mixture. C. E. RICE (U.S. Pub. Health Rep., 1925, **40**, 1808—1811).—The cyanogen chloride and hydrocyanic acid developed from sodium cyanide, hydrochloric acid, and sodium chlorate (4 oz., 17 oz., and 3 oz. respectively per 1000 cub. ft.) will kill practically all croton bugs (*Blatella germanica*—the commonest cockroach found on ships) and their eggs in 2 hrs.

R. E. THOMPSON.

PATENTS.

Process of fumigating. W. S. LANDIS and G. H. BUCHANAN, Assrs. to AMERICAN CYANAMID Co. (U.S.P. 1,559,892, 3.11.25. Appl., 8.7.21).—After fumigating the hydrocyanic acid is decomposed by the introduction of a mixture of a volatile aldehyde an ammonia.

C. O. HARVEY.

Purification of air containing ammonia. G. ST. J. PERROT and M. YABLICK (U.S.P. 1,559,930, 3.11.25. Appl., 10.1.19. Renewed 24.7.25).—Air vitiated with a high concentration of ammonia is rendered suitable for breathing by passage through a granular mass containing crystals of hydrated copper sulphate (*cf.* J., 1920, 105 A).

C. O. HARVEY.

Insecticide. G. E. SEIL and O. F. HEDENBURG, Assrs. to F. O. MOBURG, and ROESSLER and HASSLACHER CHEMICAL Co. (U.S.P. 1,559,961, 3.11.25. Appl., 28.5.21).—A 1% or more dilute aqueous solution of acetone cyanohydrin is claimed as an insecticide.

C. O. HARVEY.

Disinfectants. ERDÖL- U. KOHLE-VERWERTUNG A.-G., and F. ZERNIK (G.P. 416,599, 9.10.21).—Disinfectants are obtained by neutralising solutions containing creosote from lignite tar with naphthenic acids or sulphonated naphthenic acids. The products, which are diluted with water to about 30% creosote content, yield on further dilution stable emulsions possessing good disinfecting properties.

L. A. COLES.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 22, 1926.

I.—GENERAL; PLANT; MACHINERY.

Source of error in the mechanical analysis of sediments by continuous weighing. J. R. H. Coutts and E. M. Crowther (Trans. Faraday Soc., Nov., 1925. Advance proof).—Comparison has been made of the results obtained with the Odén–Keen balance when the shape, size, and position of the pan are altered. The low density of the suspension immediately below the pan after the sedimentation has proceeded for a few minutes sets up a flow of liquid which interferes with the free vertical fall of the particles; a direct consequence is that the observed yields are too low with narrow-rimmed pans. The magnitude of the disturbance varies with the particle size and produces a distortion of the distribution curve. Slight changes in the position of the pan when close to the base cause considerable changes in yield. F. G. Soper.

Simple method of obtaining the size distribution of particles in soils and precipitates. D. Werner (Trans. Faraday Soc., Nov., 1925. Advance proof).—The method is applicable to particles (diameter 10 to 150μ) which are too large to be measured by the automatic recording balance. A glass tube about 1·5 cm. in diameter and 1·5 m. in length is filled with the suspension, and the velocity with which the height of the sediment increases is taken as a measure of the velocity of sedimentation. Readings are taken every few seconds in the initial stages. The size distribution can be calculated from the accumulation-time curve according to the theory of Odén (Proc. Roy. Soc. Edin., 1924, 44, 98). The method was tested by means of sand particles, graded into known sizes by sieving, and good agreement was obtained between the calculated and actual size distribution. The size distribution of a clay and of precipitated strontium sulphate were also determined. F. G. Soper.

Use of metal filters for the removal of dust from air. M. Berlowitz (Gesundheitsing., 1925, 48, 397—403; Chem. Zentr, 1925, 96, II., 2075—2076).—The metal filter used in place of cloth filters for air filtration during the war is an extremely sensitive piece of apparatus, its efficiency varying with the load and the dust content of the air. It is suitable for use when the total dust content of the air to be treated is at least 1 mg. per cub. m., and when an efficiency of dust removal equal to 80% is sufficient. Should a higher efficiency be required, the metal filter is used as a pre-filter to a cloth filter. J. S. G. Thomas.

Patents.

Vessels in which solids are suspended in liquids for lixiviation, crystallisation, and the like. Aktieselskapet Krystal, Assees. of A./S. de Norske Saltverker (E.P. 219,301, 7.7.24. Conv., 17.7.23).—A layer of material placed on the perforated bottom of a vat is maintained suspended in a liquid passing up through the perforations, which are so arranged that at least one vertical section through the bottom comprises at least two perforations and further that S/F is less than $1/[5-b/l+(a-3)/7]$, where S is the area of a circular hole with well-rounded edges and with the same resistance as the perforations, F is the greatest cross-section of the suspension compartment perpendicular to the flow, b the average width and l the average length of the least cross-section of the perforations, and a the average distance in cm. between their longitudinal axes. Under these conditions the granules of the material are maintained in continuous relative movement. S. Binning.

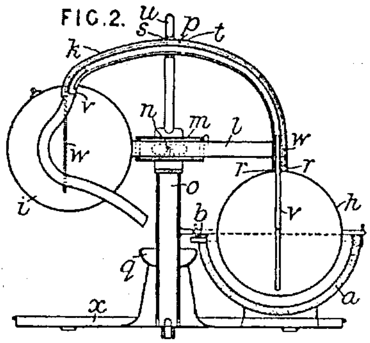
Apparatus for concentrating and cooling aqueous liquids and producing therefrom crystals of ice or other substances. P. J. de St. Andre Meynardie (E.P. 232,589, 8.4.25. Conv., 16.4.24).—The solution is sprayed unto a high vacuum at the top of a barometric column where it is cooled by rapid evaporation, the crystals and liquid are caught in a receiver at the bottom of the column, which is open to the atmosphere, and from the receiver are pumped through a filter which separates the crystals not retained in the receiver, the liquid passing on to be again sprayed. The water vapour produced at the top of the column is absorbed by a liquid absorbent sprayed into an adjoining barometric column, and from the bottom of the latter column the bulk of the diluted absorbent is pumped through a cooler back to the absorption spray, but a part is passed through a heat interchanger where it is warmed and then down a tower or other contact apparatus where it meets a stream of hot gases of combustion which dry it. The hot dry absorbent is then cooled first in the heat interchanger and afterwards in the same cooler as the main stream, or in another cooler, and united with the main stream. The hot gases from the dryer may interchange their heat with the ingoing air for combustion. B. M. Venables.

Crystallisation. H. Howard, Assr. to Grasselli Chemical Co. (U.S.P. 1,559,703, 3.11.25. Appl.,

5.7.23).—By the introduction of a small stream of supersaturated solution of the material to be crystallised into an ascending current of the solution a supply of seed crystals is maintained, and on them material from the solution is deposited, causing the crystals to grow. H. Moore.

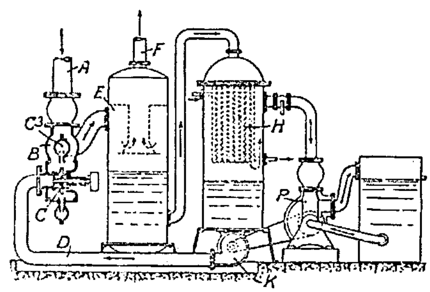
Absorption refrigerating machines. Gebr. Bayer (E.P. 234,110, 13.5.25. Conv., 16.5.24).*—The boiler-absorber, *i*, and the condenser-evaporator, *h*, are both spherical and are connected by a cross-arm, *l*, mounted on pivots, *n*, and also by a group of

FIG. 2.



curved channels or pipes, *k*, *v*, *w*, the pipe, *k*, being divided by a partition, *p*, and provided with two water inlets, *s* and *t*. In the position shown in the figure the vessel, *h*, contains the volatile liquid from which vapour is drawn through the channel, *w*, by reason of the vacuum produced in the other vessel, *i*, by means of the cooling water flowing in at *s* and through the left-hand side of the conduit, *k*. The vessel, *i*, contains absorbing liquid, through which the volatile fluid bubbles from the conduit, *w*. The evaporation in *h* causes freezing of water contained in the bowl, *a*. When this is complete the bowl is broken away by shifting and turning the device and another bowl substituted. The transfer of the volatile fluid causes the vessel, *i*, to overweigh *h*, and it drops within range of a heater (not shown) and at the same time the cooling water supplied through *u* runs into the inlet, *t*, of the right-hand section of the conduit, *w*, spreading over the spherical surface of the vessel, *h*, and being caught in the bowl, *a*. In this position the volatile fluid is evolved from *i*, through the conduit, *v*, and is condensed in *h*, until the latter vessel becomes the heavier, and drops again into the bowl, *a*.
B. M. Venables.

Separation of vapours by absorption. P. C. Lemale (E.P. 241,752, 19.12.24).*—The absorbing liquid is fed by a pipe, *D*, to the rotor, *C*, provided with a number of radial nozzles, the jets from which enter the annular intake, C^3, and create a vacuum, drawing in the gases and vapour to be washed through *A* and *B*; at the same time the spray formed absorbs the vapour. The mixture passes to a separator, *E*, whence the permanent gases pass out through *F* and the liquid solution of absorbent and vapour flows to a heater, *H*, where the absorbent is regenerated and returns by pipe, *D*, to be used



again. The recovered vapour is liquefied in the condenser, *P*, and collected in a receiver.
B. M. Venables.

Pulverisers. [Rubber lining for ball mills.] B.F. Goodrich Co., Assees. of J. R. Gammeter (E.P. 240,124, 21.5.25. Conv., 20.9.24).—A resilient lining is secured to the interior of a ball mill by means such as metal plates and bolts which are buried in the lining and are not exposed in the interior of the mill; the resilient lining is preferably thickened over the attachments, thus forming a wavy surface with the ridges running parallel to the axis. The sections of the lining overlap and are preferably formed with less curvature than that of the mill, so that when fitted the inner surface will be in compression. Preferably the resilient wearing surface of the lining is backed by a harder and cheaper material in which the means of attachment are buried, the two layers being integrally united. B. M. Venables.

Continuous crystallising apparatus. H. Howard, Assr. to Graselli Chemical Co. (U.S.P. 1,560,473, 3.11.25. Appl., 19.5.24).—The apparatus comprises a double-walled vertical vessel expanding upwards. A cooling medium is supplied to the inner vessel and the liquid to be crystallised to the annulus.
B. M. Venables.

Heating of liquids by direct contact with combustion products. Combustion reactions and their utilisation. S. C. Smith (E.P. 242,680–1, 12.5.24). — (a) Liquids are heated for evaporation or other purposes by means of products of combustion issuing from a burner directly into the liquid. The burner consists of an inner chamber of refractory material in which combustion is practically completed; the refractory material is surrounded by a pressure-tight casing and between the two are one or more annular spaces in which one or both elements of the combustion (*e.g.*, gas and air) are preheated by passing over the outer surface of the refractory material. The elements of combustion are usually supplied under pressure. The outer surface of the combustion chamber is kept cool by the incoming air, and the nozzle of the burner, which projects into the interior of the vessel containing the liquid to be heated, is reduced in size, giving a high velocity of efflux and rapid transfer of heat to the liquid, so that even rubber-like non-corrodible coatings may be used in the vessel. In addition to the vessel in which

the burner is fixed, additional means such as columns or towers may be used for further abstracting the heat from the gases. Combustion is started by means of a special torch. (B) An apparatus as described in (A) is employed to utilise the heat of combustion of substances other than ordinary fuels. For example, sulphur or hydrogen sulphide is burnt with air or oxygen and the products are injected into sulphuric acid with the double object of concentrating the sulphuric acid already formed and of using the vapours to form fresh acid. Hydrogen and chlorine may be burnt together and injected into a liquid or suspension which it is desired to treat with hydrochloric acid. B. M. Venables.

Production of colloidal suspensions. G. C. Hurrell (E.P. 242,689, 17.7.24).—To produce an emulsion of an insoluble solid, *e.g.*, sulphur, in a medium boiling below the liquefaction point of the solid at atmospheric pressure, the solid is liquefied under pressure, in communication with the dispersion liquid, *e.g.*, water containing a small amount of electrolytes or protective colloids, and the two liquids are emulsified in a homogeniser or colloid mill. The emulsion is cooled under pressure until the previously solid particles re-solidify in suspension. B. W. Clarke.

Temperature - sensitive fusible elements. H. J. C. Forrester. From Federated Engineers Development Corp. (E.P. 242,745, 1.10.24).—A non-metallic substance which remains solid substantially up to its melting point and does not become plastic like wax, resin, etc., is used as a fusible element in temperature-sensitive devices. Crystalline organic compounds selected according to the atmospheric conditions are suitable materials. B. W. Clarke.

Combined washers and dryers of the continuous centrifugal type. E. D. Eddy and A. W. Campbell (E.P. 242,829, 24.1.25).—The solid materials to be treated, *e.g.*, seeds or other granular substances, are mixed with liquid and supplied to the interior of a rapidly rotating cylindrical screen, which is divided into two sections. In the first section the liquid is removed and in the second the material is washed and dried; the wash liquor and original liquor are discharged through separate outlets. Within the cylindrical screens are conveying and scraping devices, which rotate at a slightly different speed to the screen. The conveying device consists of a number of inclined flights which do not quite touch the interior surface of the screens, and do most of the work of pushing the material forward; these are immediately followed by an equal number of scraping blades which remove the smaller material, fluff, etc., and keep the screens clear. B. M. Venables.

Non-aerating apparatus for centrifugally purifying liquids. S. H. Hall, Assr. to De Laval Separator Co. (U.S.P. 1,561,784, 17.11.25. Appl., 8.10.24).—A centrifugal separating bowl is provided with feeding means controlled so as to prevent either overflow of liquid or admission of air. The purified liquid is exhausted through the neck of the bowl to a conduit provided with means to maintain a pressure above that of the atmosphere. The neck is surrounded by a bearing which is supplied with liquid at a point near the outlet end of the neck; the liquid flows through the bearing, both downwards to an escape outlet and upwards to the main liquid discharge. Any liquid which works down below the bearing escape outlet is returned upwards to it by means provided in the bearing. B. M. Venables.

Producing catalytic reactions. H. E. Haggenmacher (U.S.P. 1,557,687, 20.10.25. Appl., 13.4.21).—The catalytic material is introduced into the top of a vertical cylinder up which the reacting gases are passed, and is withdrawn from the bottom of the cylinder, revivified if necessary, and used again. Accumulation of catalyst poisons and production of side reactions are thus prevented. T. S. Wheeler.

Material for absorbing gases, liquids, and solids, and for effecting catalytic reactions. Badische Anilin- u. Soda-Fabr., Assees. of A. Mittasch and J. Jannek (G.P. 418,032, 27.2.23).—The material is obtained by extracting humic coal, *i.e.*, lignite with a high humin content, with alkaline solutions. The solutions obtained may be used direct, or may be dried, or the material precipitated by acidifying the solutions, or solutions containing the precipitated material may be used. L. A. Coles.

Foam-stabilising-composition. G. J. Esselen, jun., Assr. to Badger Fire Extinguisher Co. (U.S.P. 1,558,599, 27.10.25. Appl., 22.4.24).—A mixture of sodium resinate (0·5 pt.) and the product (20 pts.) obtained by neutralising sulphite-cellulose waste liquor with lime and evaporating it to dryness is added to the sodium bicarbonate mixture used in foam fire extinguishers in order to stabilise the foam. T. S. Wheeler.

Separating materials of different specific gravities. T. M. Chance (U.S.P. 1,559,937–8, 3.11.25. Appl., [A] 24.1.21, [B] 7.7.22).—(A) A mixture of a liquid and insoluble comminuted solids is maintained by agitation at a density between the densities of the materials to be separated, the heavier constituent being allowed to sink out of the mixture through a rising current of the liquid beneath and not divided from the agitated mixture. (B) A mixture as above is maintained in a fluid condition by rotation of the container, *e.g.*, a drum, and by the introduction of liquid under pressure. A partition in the fluid mixture guides the floating constituent to the outlet, and means are provided for introducing the raw material and for removing the heavier constituent. B. M. Venables.

Method for producing fluid mediums of high density. T. M. Chance (U.S.P. 1,561,909, 17.11.25. Appl., 29.12.24).—A mixture of liquid and comminuted insoluble solids of greater density than the liquid is prevented from separating by supplying liquid under pressure to the lower part of the mixture, the smaller and lighter particles being thereby

continually carried up through the mixture. The upper layer thus formed is continuously removed from the containing vessel and returned to it at a level considerably below the top.

B. M. VENABLES.

Apparatus for transmission of heat from one medium to another. E. S. LYNGER (U.S.P. 1,560,271, 3.11.25. Appl., 12.6.24).—An apparatus suitable for preheating air for combustion by means of hot flue gases comprises a centrifugal blower with an axial inlet. The inlet chamber and the blower chamber are each divided into two compartments, one for gas and one for air. In one half of a revolution the vanes of the blower propel the flue gas and are heated. In the other half they propel air and give up their heat to it.

T. S. WHEELER.

Apparatus for concentrating liquids. F. L. ANTISELL (U.S.P. 1,561,898, 17.11.25. Appl., 17.2.23). —A combustion chamber is situated above a chamber containing the liquid to be evaporated, the two being separated by a horizontal partition through which vertical heat-conducting elements extend in both directions and dip into the liquid.

B. M. VENABLES.

Measurement of relative humidity [in technical drying processes]. A.-G. FÜR ANILIN-FABR., Assees. of R. FUCHS and H. WOLFF (G.P. 416,563, 3.7.24).—Relative humidity is measured by comparison of the physical constants of the outflowing air and a comparison gas. The latter is saturated with water vapour at the temperature of the outflowing air by bringing the measuring apparatus or the parts of it containing water into the stream of outflowing air, or by bringing them to the temperature of the latter by suitable heating arrangements. The end of the drying is indicated by a constant maximum difference in the property measured between the dry outflowing air and the saturated comparison air. B. FULLMAN.

Centrifugal separator. H. P. HOYLE (U.S.P. 1,563,491, 1.12.25. Appl., 12.7.23).—See E.P. 206,585; B., 1924, 40.

Rotary drying apparatus. S. S. GETTINGS and W. L. BODMAN, Assrs. to J. M. T. JOHNSTON (U.S.P. 1,564,058, 1.12.25. Appl., 27.9.24).—See E.P. 223,042; B., 1924, 999.

[Means for sealing evacuated] furnaces. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of W. F. MASSEY (E.P. 235,565, 9.6.25. Conv., 12.6.24).

Grates for shaft furnaces. E. CORNET (E.P. 243,050, 16.8.24).

[Laboratory] ovens. H. S. SHARMA and G. D. DESAI (E.P. 243,223, 14.4.25).

Cooling and moistening of air. S. YAMAMOTO (E.P. 243,061, 20.8.24).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Colloid chemistry of humic acid and peat. WO. OSTWALD and A. STEINER (Koll. Chem. Beihefte, 1925, 21, 97—170).—Humic acid preparations have been made from peat and purified by dialysis, and their diffusion, ultrafiltration, electrophoresis, surface tension (cf. A., 1925, ii, 771), and especially viscosity studied. Humic acid sols are negatively charged, moderately hydrated systems which in many respects, especially in regard to flocculation, behave like negatively charged hydroxide sols, such as molybdenum dioxide and vanadium pentoxide; for instance, with salts they, and also peat suspensions, give flocculation values in approximately the same order. The flocculation of both humic acid sols and peat suspensions by a variety of electrolytes has been studied, and is found to result in an increase in viscosity and in the velocity of filtration. This behaviour is due to the formation of larger "secondary particles," which can again unite to form aggregates of higher orders, as is shown by the great influence of previous mechanical treatment on the viscosity of the flocculated sols and suspensions. By the addition of small quantities of alkali all the flocculated humic acid preparations studied are peptised. After flocculation with salts of iron or other heavy metals, organic bases, *e.g.*, pyridine, must be used as peptising agents. Free chlorine, in the form of chlorine water, is an especially active flocculating agent. In drying peat the presence of flocculating agents, especially chlorine, results in essentially harder and denser residues, although the drying process itself may be retarded on account of shrinkage of the evaporating surface.

N. H. HARTSHORNE.

Binders for briquetting, with special reference to "pulp binders." C. J. GOODWIN and G. N. WHITE (Proc. S. Wales Inst. Eng., 1925, **41**, 443—461).—Waste vegetable matter (grass, beet sugar residues, banana leaves, etc.) is stacked and watered from time to time with a culture solution for bacteria. After the lapse of a variable period fermentation produces a pulpy matrix of humus substances intercalated with residual fibres. This may be treated in a suitable mill, if necessary with a small quantity of weak alkali. The hardening and agglomerating properties are developed by heating the briquettes to 100° or, if additional waterproofness is required, to 200°, when the material approaches pitch in waterproofing properties. Tests on strength and resistance to weathering of briquettes made with this binder gave favourable results. Combustion trials in open stoves and grates with strong draught have been completely successful. Cost and space requirements for production are discussed. The substance is applicable where coal is friable or where washery slurries and semi-coke breeze are produced.

R. A. A. TAYLOR.

Calorimetric bomb. P. LANDRIEU (Bull. Soc. chim., 1925, [iv], **37**, 1340—1359; cf. Moureu,

B., 1925, 301).—The chief errors in the ordinary Berthelot calorimetric bomb are due to loss of heat by evaporation of the water, gain of heat from the effect of the stirrer, and to differences of heat exchange in different experiments. A new bomb is described in which the water container is hermetically sealed, and the heat exchange regulated by always starting at the same temperature. The heating effect of the stirrer is proportional to the cube of the velocity and its effect can be determined. W. Hume-Rothery.

Bergius process of converting coal into oils. F. Bergius (Z. Ver. Deuts. Ing., Oct. 17 and 24, 1925; Engineering, 1925, 120, 675).—A continuous plant at Rheinau, near Mannheim, for the hydrogenation of coal has been in operation for more than a year, and deals with 5 tons daily at an initial pressure of 120 atm., which is to be raised to 150 atm. A paste of coal ground to 1 mm. size, with 40% of tar (freed of its highly volatile constituents) is forced into the iron reaction chamber, which is heated indirectly by compressed, preheated inert gas in the jacket surrounding the chamber; external heat is provided by means of a lead bath. One ton of coal yields 445 kg. of oil (rich in cresols and phenols), 210 kg. of gas, 5 kg. of ammonia, and 350 kg. of residual carbon. A. C. Monkhouse.

Distillation of cellulose, wood, and similar materials in the presence of hydrogen under pressure and catalysts. H. E. Fierz-David and M. Hannig (Helv. Chim. Acta, 1925, 8, 900—923).—The dry distillation of cellulose, wood, starch, or lignite appears not to be affected by the presence of hydrogen at 300 atm. pressure. With nickel oxide and hydrogen together (yielding finely-divided nickel), however, under high pressures, complete decomposition of these substances to liquid and gaseous products is effected. Copper is much less effective and iron ineffective owing to the fact that reduction is not complete until the materials are largely decomposed. Gas-coals yielded unfavourable results. The most important products which have been isolated are aromatic phenols (homologues of xylenols, guaiacol), liquid organic acids from formic to valeric acids, numerous diketones, few ketones, numerous alcohols including methyl alcohol, a cyclic glycol, several homologues of furane, and carbon monoxide, carbon dioxide, and water. Details are given concerning the gas compressor, preparation of materials, and the analysis of the liquid and gaseous products. R. A. Morton.

Thermal decomposition of cellulose under hydrogenation conditions. A. R. Bowen, H. G. Shatwell, and A. W. Nash (J. S. C. I., 1925, 44, 507—511 T).—The decomposition of cellulose at temperatures up to 440° in the presence of hydrogen at pressures of 120 atm. resulted in the formation of gas, aqueous liquor, a small quantity of tar, and solid residue, no appreciable amount of hydrogen being absorbed. However, in the presence of 1% of nickel oxide under similar conditions, 3—4% of hydrogen was absorbed and the products consisted of gas, aqueous liquor, tar, and only a trace of solid carbonaceous matter. The tar (22·4% of the weight of cellulose used) contained about 13% of a brown powder insoluble in ether, no carboxylic acids, 2·3% of phenols, and 77% of neutral oil. Vanadium salts have but slight catalytic influence in this reaction.

Direct hydration of aliphatic carbon, and the assimilation of carbon dioxide by plants. F. J. Nellensteyn (Chem. Weekblad, 1925, 22, 565—571).—Wood-charcoal and carbon black are readily oxidised by permanganate, with formation of carbon dioxide and of acidic substances. The latter may be isolated by means of their barium and lead salts, and are hygroscopic, syrupy substances with caramel-like odour, soluble in alcohol and water, but not in ether etc. Analyses of the salts and the free acids indicate that the latter are dicarboxylic hydroxy-acids of the sugar acid group. Norit on similar treatment is less vigorously attacked, but the acids obtained contain mellitic acid and yield benzene on dry distillation with sodium hydroxide; this charcoal is therefore a mixed aromatic-aliphatic carbon, whereas wood-charcoal and carbon black are aliphatic carbons. The nature of the products obtained from these latter indicate that hydration of the carbon atoms has occurred simultaneously with the oxidation, since the products are similar to those obtained by the oxidation of carbohydrates (sugars and polyhydric alcohols). The reactivity of finely-divided carbon, and the formation of carbohydrates by hydration of this material, afford an explanation of the assimilation of carbon dioxide by plants, if it be assumed that reduction of carbon dioxide in the plant tissue gives rise to aliphatic carbon, which then immediately becomes hydrated. S. I. Levy.

Identification of "norit" and other wood charcoals. H. G. Tanner (Ind. Eng. Chem., 1925, 17, 1191—1193).—Microscopical and macroscopical examination of "norit" decolorising carbon indicates that it is made from birch-wood (*Betula occidentalis*), probably by carbonisation of the sawdust of the wood in the presence of a limited amount of air, followed by acid extraction, washing, and drying. It is possible to identify a considerable number of wood charcoals microscopically by means of the character of the perforations in the particles. J. P. Ogilvie.

[Gas] purification. T. R. Cook (Gas J., 1925, 172, 432—437).—In the purification of gas at Southport the inadequacy of the ordinary scrubber system (a Livesey washer, a P. and A. tar extractor, a Holmes and a Clapham washer-scrubber) threw an overload on the oxide purifiers. The American system of washing the gas with 3% soda ash solution was resorted to. A scrubber with a spray and wooden grids was inserted before the oxide boxes. The resulting liquor was revivified by passing it through a second similar scrubber up which air was blown. During gas scrubbing hydrogen sulphide, hydrogen cyanide, and carbon disulphide are removed with the formation of sodium hydrogen sulphide, and

sodium cyanide and thiocarbonate. During revivification these are oxidised to sodium polysulphide and thiocyanate and sodium salts of oxy-acids of sulphur. The revivified liquor is spent when its sodium thiocyanate content reaches 10%. The air used in revivification is passed through an open purifier as it is fouled with regenerated hydrogen sulphide. This washing process, besides reducing the overload on the oxide purifiers, increases their efficiency by reducing the amount of tar fog which fouls them. It reduces incidentally the naphthalene content of the gas. The cost is 0·4d. per 1000 cub. ft., and the quantities of materials required per 1000 cub. ft. of gas purified are 0·08 lb. of soda ash, 65 gals. of solution, and 66 cub. ft. of air for revivification. The hydrogen sulphide in the gas between the Clapham washer-scrubber and the oxide boxes was reduced by nearly 50%. R. A. A. TAYLOR.

Purification of coal gas by the wet process. C. BERTHELOT (Chim. et Ind., 1925, **14**, 663—678).—The development of the utilisation of coke-oven gas as town gas in France raises the question of the purification of the gas before transport. The American process in which the impurities are absorbed in a solution of sodium carbonate, containing iron oxide in suspension, which is revivified by a current of air, is the most economical and suitable method available. A full account of American practice is given, and a design for a complete purifier plant is suggested. A. B. MANNING.

Gas analysis. P. LEBEAU (Chaleur et Ind., 1925, **6**, 236—245; Chem. Zentr., 1925, **96**, II., 2071—2072; cf. Lebeau and Bedel, B., 1924, 772).—Acetylene is absorbed in an alkaline solution of potassium mercuric iodide prepared by dissolving 25 g. of mercuric iodide and 30 g. of potassium iodide in 100 c.c. of water. Potassium hydroxide is added at the time of use of the solution in the absorption apparatus. 1 c.c. of the solution absorbs 20 c.c. of acetylene. The absorption of ethylene by concentrated sulphuric acid is accelerated by the addition of about 1% of the acid anhydride of vanadium, tungsten, molybdenum, or uranium, which after reduction by electrolysis or by contact with mercury, aluminium, or zinc exerts a catalytic effect, so that ethylene is absorbed after shaking for less than 1 minute. Homologues of ethylene are similarly absorbed: members of the CH_4 series are not absorbed. By appropriate cooling with liquid air, carbon dioxide snow, or acetone etc. over mercury it is possible to separate the higher hydrocarbons of the series C_nH_{2n+2}, C_nH_{2n}, and C_nH_{2n-2}, as well as carbon dioxide, hydrogen sulphide, ammonia, etc., from the hydrogen, methane, carbon monoxide, oxygen, and nitrogen, which remain gaseous at low temperatures. In this manner the fractionated hydrocarbon content of a gaseous mixture is further fractionated at appropriate temperatures into portions containing not more than two homologues, the proportions of which may be determined eudiometrically. The fractionation of a mixture containing ethane, propane, and butane at temperatures between $-135°$ and 14° is detailed. J. S. G. THOMAS.

Determination of water in hydrocarbon oils, shales, and lignites. C. E. WOOD and A. E. T. NEALE (J. Inst. Petrol. Tech., 1925, **11**, 471—474).—A sufficient quantity of oil to give less than 0·5 c.c. of water is distilled with toluene or xylene in a flask of 150 c.c. capacity provided with a bent tube leading to a measuring tube immersed in a freezing mixture, till 20 c.c. of distillate are collected. Aniline is added to the distillate until critical solution is obtained at 20°. The amount of aniline used corresponds to the amount of water in the distillate, and this may be read off from graphs prepared for various solvents. The results with benzene as a solvent were not satisfactory. The method may be applied to the determination of water in solids. In each case the results were somewhat higher than those obtained by drying in a vacuum. A special method of determination is necessary in the case of kerosene or petrol, as lower boiling fractions distil over with toluene or xylene, and results are obtained by comparing the aniline required with that required for dried oil to which a given quantity of water has been added. H. MOORE.

Toxic gases from Mexican and other high-sulphur petroleums and products. R. R. SAYERS, N. A. C. SMITH, A. C. FIELDNER, C. W. MITCHELL, G. W. JONES, W. P. YANT, D. D. STARK, S. H. KATZ, J. J. BLOOMFIELD, and W. A. JACOBS (U.S. Bureau Mines Bull. 231, 1925, 108 pp.).—Hydrogen sulphide is the only toxic gas, apart from petroleum vapour, evolved from Mexican and other petroleums with a high sulphur content. Acute hydrogen sulphide poisoning, resulting in respiratory paralysis followed by heart failure, can be produced by concentrations of the gas above about 0·06—0·08%, and sub-acute poisoning in the form of irritation of the eyes, nose, pharynx, and bronchial passages, can be produced by prolonged exposure to concentrations as low as 0·005%. During the distillation of light Mexican crude oil containing about 4% S the greater part of the sulphur collects in the gases, and in the heavier fractions and the residuum. A large percentage of the sulphur is combined in the form of a black, brittle asphalt, which is insoluble in acetone, but soluble in benzene, but even after removal of the asphalt, the residual oil has the characteristics of a high-sulphur crude. Heavy Mexican crudes do not give off such large quantities of hydrogen sulphide as the light crudes. Evolution of hydrogen sulphide is almost continuous during the handling and working up of high-sulphur crudes, and in general the concentration of hydrogen sulphide in the gases evolved is successively lower in the crude stills, in the pressure stills, and in tankers and other containers for the crude oil. Gases in containers for finished products are free from hydrogen sulphide. An account is given of the general methods of producing, transporting, and refining light Mexican crude oils, and of methods adopted for safeguarding

employees at places where they are liable to be overcome by the gas. L. A. Coles.

Paraffin wax and its properties; methods of testing wax, and of analysing oil-wax mixtures. L. D. Wyant and L. G. Marsh (U.S. Bur. Mines, Tech. Paper 368, 1925, 26 pp.).—The methods of production of lubricating oils and the separation of paraffin wax from petroleum are described. The press method of determining the oil content of wax is unsatisfactory for wax-oil mixtures containing more than 10% of oil. The A.S.T.M. method for melting point was found to be very accurate: the Petrolatum melting point method of the A.S.T.M. was used in the experiments. For the separation and determination of wax, acetone was used as a solvent, 5 g. or 10 g. of the sample being dissolved by heating under a reflux condenser in enough acetone to make 100 c.c. of solution. The solubility of wax fractions in acetone does not vary greatly between 10° and 21°, the solubility being more affected by the melting point of the wax than by differences of temperature between these limits. Wax recrystallised from acetone is more nearly free from oil than ordinary commercial wax. The accuracy of the method increases with the percentage of wax in the sample and its melting point. H. Moore.

Recovery of sodium plumbite in the oil industry. A. Kinsel (Chem. Met. Eng., 1925, 32, 873—874; cf. U.S.P. 1,525,301, B., 1925, 276).—The lead sulphide and spent caustic soda from the "doctor" treatment are run off to a settling tank heated with a steam coil. From this the caustic solution is drawn off to storage and the lead sulphide has its water content reduced to 30% in a continuous filter. The sulphide is then treated with sludge acid diluted to 35—40° B. (*d* 1·32—1·38) and converted into lead sulphate. This, mixed with caustic soda solution, generates fresh "doctor" solution (sodium plumbite). 99% of the lead is thus recovered, and 75—80% of the caustic soda, and the sludge acid is partially utilised. The gasoline drawn off with the lead sulphide is recovered. The lead sulphate is more easily soluble than litharge, and danger of lead poisoning is avoided. A plant to recover 500 lb. of litharge per day costs $6000, and operating and fixed charges are about $5.00 per day. H. Moore.

Studies on naphthenic acids. VI. Naphthenic acids derived from Nishiyama petroleum. Y. Tanaka and S. Nagai (J. Fac. Eng. Tokyo, 1925, 16, 171—181).—Crude naphthenic acids from a kerosene distillate, *d* 0·817, obtained in refining Nishiyama petroleum, closely resemble, and contain the same constituent acids as those derived from a neutral oil distillate from the same source (cf. B., 1925, 870). L. A. Coles.

Spreading of lubricants on metallic and solid surfaces. P. Woog (Compt. rend., 1925, 181, 772—774).—The behaviour of oils on a surface of liquid mercury resembles that on water (Woog, Compt. rend., 1922, 174, 162). On clean surfaces of steel, brass, and agate, drops of vaseline oil, containing only saturated molecules, rapidly cover the whole surface, whereas drops of olive oil remain as globules owing to the attraction between the surface and the oriented active unsaturated molecules. Machinery oil containing a mixture of saturated and unsaturated molecules shows an intermediate effect. The spreading effect of inactive saturated mineral oils can be neutralised by adding small quantities of active substances such as cetyl alcohol or oleic acid; by coating the metallic parts concerned with a thin film of active substance, clocks can be lubricated with pure mineral oil, which is unsatisfactory under ordinary conditions owing to the spreading effect. W. Hume-Rothery.

Transformer oils. Musatti and Pichetto.—See XI.

Determining sulphur in mineral oils. Wagner.—See XX.

Sewage for gas production. Sierp.—See XXIII.

Patents.

Method and apparatus for complete combustion. E. Gates (U.S.P. 1,560,076, 3.11.25. Appl., 8.4.22).—Finely-divided solid or liquid fuel or a mixture of the two is passed with just sufficient air for complete combustion through a helical passage of resistant material which is thermally insulated. The walls of the passage beome incandescent, and promote the combustion of unburnt particles which strike against them. This will occur frequently as the unburnt particles are heavier than the gaseous products of combustion. T. S. Wheeler.

Manufacture of fuel briquettes. L. A. Wood, and Minerals Separation, Ltd. (E.P. 242,352, 6.8.24).—The production of smoke on burning moist briquettes is eliminated by heating the briquettes in superheated steam at 100—300°, whereby the smoke-producing materials are removed. The briquettes are made by flocculating a suspension of the fuel in water with a carbonaceous binder, such as pitch or tar, and compressing the product. R. A. A. Taylor.

Manufacturing smokeless briquetted fuel from small coal. Midland Coal Products, Ltd., and C. Ingman (E.P. 242,783, 7.11.24).—In a modification of the process described in E.P. 198,503 (J., 1923, 701 A), suitable mixtures of finely divided non-caking and caking coals are compressed into briquettes of spheroid or ovoid shape, using pitch as a binder, and carbonised in a continuous vertical retort. Carbonisation is effected by the combustion of a portion of the briquettes by admitting air or steam at the base of the retort. A. C. Monkhouse.

Carbonising ovoid briquettes in a vertical retort. E. Gevers-Orban (E.P. 242,869, 6.4.25).—Ovoid coal briquettes are carbonised in a continuous vertical retort with heating flues arranged to give the

maximum heat at the top of the retort, thus hardening rapidly the outer surface of the briquettes. The gases resulting from the distillation are used for heating the combustion chambers and also for the recovery of heat from the briquettes by passing a portion of the gases through the charge.

A. C. MONKHOUSE.

Distilling bituminous substances at a low temperature. KOHLENSCHEIDUNGS GES. (E.P. 225,172, 6.8.24. Conv., 24.11.23).—Bituminous substances are subjected to low-temperature distillation in two stages. The material is first heated in a rotary retort to a temperature which does not rise considerably above the softening point of the material, *e.g.*, for most coals not above 400°. It then passes directly into a vertical retort, where the carbonisation is completed. The material is conveyed along the rotary retort to avoid caking, but when in the vertical retort is moved as little as possible. The production of dust is thus reduced to a minimum.

A. B. MANNING.

Method and means for stimulating distillation. J. J. DESCHAMPS (E.P. 234,840, 29.5.25. Conv., 30.5.24).—The reactions of the distillation products in the presence of the solid material in coke ovens, gas retorts, etc., are stimulated by imparting pulsations to the vapours. This is effected by inserting in the pipe leading to the tar main an apparatus which works intermittently on the principle of an ordinary laboratory filter pump and alternately relieves the pressure and allows it to accumulate. The device is operated by the purified distillation gases, or by an auxiliary gas.

R. A. A. TAYLOR.

Coke ovens. E. COPPÉE ET CIE. (E.P. 235,546, 22.5.25. Conv., 12.6.24).—A series of apertures, which can be opened or closed by means of hinged bricks, is provided above the walls dividing the heating circuits in a coke oven, so that when closed the wall flues are grouped into a number of independent circuits, each consisting of one or more ascending flues and one or more descending flues. This forms the normal working arrangement of the oven. The apertures are opened to allow intercommunication between the heating circuits for the distribution of the drying smoke during the period of preheating.

A. B. MANNING.

Coking retort ovens. P. C. RUSHEN. From KOPPERS Co. (E.P. 241,991, 30.7.24).—A coking oven is provided with a large number of ports in the bottom wall of the chamber, between the longitudinal centre line and the sides, through which steam is injected during the later part of the coking period. The number and position of the ports are such that the steam effectively penetrates the coked portion of the charge. The steam channels are so disposed relatively to the passages conveying hot gases to the regenerators that the steam is superheated before admission to the oven.

A. B. MANNING.

Distillation of fuel. A. J. A. HÉRENG (E.P. 242,411, 11.10.24).—The gaseous products of distillation are burned in a combustion chamber so that nitrogen and one of the oxides of carbon, but no free oxygen result. These gases are passed into the retort to effect distillation. The gas leaving one retort passes on to other retorts to preheat fresh charges.

R. A. A. TAYLOR.

Distillation of coal. J. NEATH and W. CHANEY (E.P. 242,435, 8.11.24).—A setting of retorts for low-temperature distillation is built with a regenerator and gas producer so that the blow-gas from the producer passes through the regenerator in which secondary air ports admit air to burn it; it then passes on to heat the retorts externally. The water-gas made then passes through the regenerator with the air ports closed, and is used for heating the retorts internally.

R. A. A. TAYLOR.

Production of combustible gases. H. A. HUMPHREY, and SYNTHETIC AMMONIA AND NITRATES, LTD. (E.P. 242,741, 24.9.24).—Combustible gases are produced from powdered fuel by causing the latter to react with highly preheated steam and air. The combustion zone is maintained at about 1300°, and the preheated gases enter the combustion chamber at about 1000°. A gas consisting only of carbon monoxide, hydrogen, and nitrogen is produced, the formation of carbon dioxide or hydrocarbons being precluded by the temperatures used. The high degree of preheating is attained by the use of special regenerators of large dimensions. A working unit consists of two combustion chambers and two regenerators. While one combustion chamber is in operation, the hot gases therefrom pass through the other and thence through the corresponding regenerator. It is preferable to use sufficient steam to ensure complete gasification of the fuel with the minimum quantity of air. If a gas rich in hydrogen is required, the carbon monoxide in the product is caused to react with steam: $CO+H_2O=CO_2+H_2$, and the heat from this exothermic reaction may be made to contribute to the maintenance of the high furnace temperature.

A. B. MANNING.

Process for splitting coal, oils and other hydrocarbons, by heating these initial materials with hydrogen under high pressure. E. C. R. MARKS. From INTERNAT. BERGIN-COMP. VOOR OLIE EN KOLEN-CHEMIE (E.P. 242,876, 27.4.25).—In the hydrogenation of oils, coal, etc., at pressures of 100—150 atm., and temperatures of 300—500°, where it is desired to feed the material continuously into the reaction chamber in the form of a paste, it is first compressed by means of a worm conveyor up to a few atmospheres pressure, and then forced by a hydraulic press into the reaction vessel. A definite liquid level is maintained in the reaction vessel by drawing off the reaction products through a pipe that dips down to the required normal level.

A. B. MANNING.

Retort resembling a converter, for distilling material containing hydrocarbons. M. DEVAUX (G.P. 414,489, 14.2.24. Conv., 4.2.24).—A cylindrical or prismatic retort of great length in proportion to its cross-section, rotates about its transverse axis.

The heating gases enter the retort through one of the trunnions, which are hollow, and are distributed throughout the charge by means of perforated tubes extending to the retort walls The distillation products and the spent heating gases pass together into a chamber extending the whole length of the retort, and pass out of the retort through the other trunnion. L. A. Coles.

Production of low-temperature tar. E. Weiss (G.P. 417,688, 21.1.21. Can. P. 234,825, 24.1.22).—Semi-coke, gas, and low-temperature tar, are obtained by heating solid fuel in a rotating retort, with a counter-current of the combustion gases obtained by burning waste coal, the gases being reduced to a temperature suitable for the production of low-temperature tar, by admixture with non-combustible gas, before entering the retort. L. A. Coles.

Producing oil-gas in externally heated retorts. J. Rude (E.P. 242,029, 15.9.24).—Water-gas or other gas produced in externally heated vertical retorts is enriched by oil-gas produced within the retort. The oil is injected into the retort, and provision is made to remove the oil-gas directly it is formed. The oil-gas residue forms a valuable addition to the coke. The process can be worked in conjunction with that described in E.P. 235,007. A. B. Manning.

Gas producers. F. H. Waite and G. W. Davey (E.P. 242,473, 29.12.24).—The lining of a producer is provided with horizontal and vertical passages and a manifold for conducting air, steam, or water through these passages, whence it passes to the interior of the producer. An auxiliary conduit permits the water or steam to be conducted to the passages at different levels. The lining is thereby cooled and its life prolonged. Clinker formation on the sides of the producer is avoided and radiation loss reduced. A. B. Manning.

Eliminating hydrogen sulphide and other noxious impurities from fuel gases, and from air. W. J. Mellersh-Jackson. From Koppers Co. (E.P. 240,891, 5.6.24).—Hydrogen sulphide and other impurities in fuel gases are removed by absorption in an alkaline liquid, *e.g.*, a solution of sodium carbonate, which can be regenerated by aeration, the impurities being transferred to the stream of air by which aeration is effected (*cf.* U.S.P., 1,389,980 and 1,390,037; J., 1921, 762 A). The volume of air used is greater than that of the gas from which the hydrogen sulphide is extracted. The hydrogen sulphide is then removed from the air stream by washing in a dilute alkaline solution, *e.g.*, 0·5—1% sodium carbonate, containing 2—3% of iron oxide in suspension. The sulphur formed in this wash liquid is recovered. A. B. Manning.

Rotary scrubbers and washers for gas. Gas Light and Coke Co., and E. W. Eve (E.P. 242,404, 7.10.24).—A rotary gas scrubber has bundles of wetted elements arranged to fill practically the entire space between the central shaft and the inner periphery of the casing. The gas passes parallel to the axis of the machine, The outer elements of the bundles form pockets with the solid outer plates, so as to pick up the liquid, carry it up, and distribute it over the bundles. A. B. Manning.

Gaseous-liquid contact apparatus. [Centrifugal gas washer.] P. M. Kuehn, Assr. to Bartlett-Hayward Co. (U.S.P. 1,561,000, 10.11.24. Appl., 10.1.24). Liquid is thrown outwards from a pan over a baffle extending inwards from the casing wall, and a portion of the liquid is returned direct to the pan without passing over the baffle. The gas is introduced into the casing. S. Binning.

Distillation of [benzol] wash oil. F. Goldschmidt (G.P. 417,340, 31.7.24).—Benzene and toluene are recovered from crude light oil separated from wash oil, by heating the oil by means of wash-oil flowing out of the washing apparatus, and xylene and solvent naphtha are recovered by treating it with steam in the same or in another extraction apparatus. The residual oil is returned to the supply of wash-oil, to prevent it from thickening. L. A. Coles.

Gas and vapour indicating device. S. Ruben (U.S.P. 1,559,461, 27.10.25. Appl., 28.5.21).—An electric current is passed under a fixed voltage through a layer of palladium black or platinum sponge mounted on an insulator. If the material is in equilibrium in an atmosphere and this changes in any manner, the composition of the adsorbed gas will alter, with a consequent change in the resistance of the adsorbing layer and in the current passing through it. T. S. Wheeler.

Gas analysing method and apparatus. J. R. Cain (U.S.P. 1,560,660, 10.11.25. Appl., 3.6.19. Renewed 1.4.25).—The quantity of a substance in a mixture is determined by measuring the change in resistance produced in an electrolyte before and after adding the mixture to it, the electrolyte being one which will react with the substance in question to produce a precipitate. For example carbon dioxide is absorbed in barium hydroxide solution (cf. Cain and Maxwell, J., 1919, 773A). M. E. Nottage.

Apparatus for continuously measuring the combustible constituents of waste gases. M. Moeller, Assr. to Siemens u. Halske A.-G. (U.S.P. 1,562,243, 17.11.25. Appl., 22.11.24).—The waste gases pass through a compartment in which the combustible constituents are burnt by contact with a wire made of an alloy of platinum or other metal of that group. The electrical resistance of the wire is compared with that of a similar wire in a second compartment filled with a comparison gas. A. B. Manning.

Extraction of oil-shale, oil-sand, and similar material. G. Baume, and Soc. Recherches et Perfectionnements Ind. (F.P. [A] 589,710, 16.1.24, and [B] Addn. thereto 29,373, 11.6.24).—(A) The material is extracted with aqueous solutions containing small quantities of emulsifying agents,

such as soaps, alkali resinates, or similar substances, which affect the surface tension of the solution, preferably with the addition of substances capable of dissolving petroleum, such as trichloroethylene, or of causing agglomeration of the oil particles, such as powdered charcoal. (B) The solutions described in (A) may be acid or alkaline, and are maintained at as low a temperature as is practicable. The material is macerated with a similar solution before it is charged into the extraction bath. The residues are suitable for use in road making.

L. A. COLES.

Storing explosive gases in containers. NORDDEUTSCHE ACETYLEN U. SAUERSTOFFWERKE A.-G., and J. POMMÉE (E.P. 241,468, 27.4.25).—A mineral having absorptive properties is heated until fritting takes place and then granulated. Pieces between 2 and 3·5 mm. in size are then packed into a container prior to the introduction of a solvent fluid for the gas. The spaces between the pieces may be packed with a powdered substance such as pumice.

H. HOLLINGS.

Oil cracking stills. SINCLAIR REFINING CO., Assees. of J. E. BELL (E.P. 231,840, 21.2.25. Conv., 1.4.24).—To avoid overheating of the oil in tubular cracking stills, and consequent overheating of the tubes with risk of rupture, the incandescent fuel in the fire-box is shielded to prevent direct radiation on to the tubes. Air preheated by the hot waste gases is introduced into a throat leading from the fire-box to the heating chamber containing the tubes for the purpose of moderating the heat of the fire-gases; the efficiency of heating is thereby little diminished, or may even be increased. The oil in the tubes flows in counter-current to the gases in the heating chamber, from which the gases pass downwards into an arrangement for preheating the air supply. The preheated air is not used for purposes of combustion.

H. MOORE.

Obtaining petrolatum oils, petrolatum, and paraffin. F. LAVIROTTE (E.P. 240,803, 25.8.25. Conv., 4.10.24. Not yet accepted).—Petroleum residues and heavy oils are purified by treatment with strong sulphuric acid at an elevated temperature. Preferably the material is first treated below 100° and then at not over 200°, *e.g.*, 160—170°. Sulphur trioxide may be added to the acid, and a hydrocarbon that is liquid at the temperature used and not acted upon by the acid may be mixed with the crude material. The product is neutralised by treatment with calcium carbonate and filtration through porous clay. Oil that has been treated once with acid as above may be freed from sulphonated compounds by extraction with alcohols, light naphtha, or other solvent of the pure oil, or by further treatment with 20% oleum. For example, black cylinder oil is mixed with an equal weight of petroleum jelly that has already been treated with acid, and agitated with 1·5 pts. by weight of 20% oleum at 120°—160° for 24 hrs. The liquid is drawn off, neutralised by agitation at 120° for 3—4 hrs. with 10% of porous clay and 1·5% of calcium carbonate, and filtered hot through porous clay. The carbonaceous residue in the acid-treatment vessel is broken up and treated with naphtha, benzol, tetrachloroethylene, or other solvent of the purified oil. The solution is withdrawn, the solvent distilled off, and the oil neutralised and filtered. Alternatively, the solution is chilled and the precipitated oil or jelly allowed to settle out. The carbonaceous residue is afterwards heated to about 400° to remove volatile impurities, ground in a ball-mill, and maintained red hot in contact with air until about half is consumed. A highly adsorbent carbon is thus obtained. Sulphur dioxide evolved during the operations is absorbed in sodium carbonate solution.

Avoiding shock chill in precipitating substances [wax] from liquids [petroleum]. L. D. JONES, Assr. to SHARPLES SPECIALTY CO. (U.S.P. 1,558,619, 27.10.25. Appl., 5.2.21).—Brine is circulated through coils in a tank containing petroleum, and a portion of the brine is also circulated through a refrigerating system, so that the temperature of the brine is maintained about 5° below that of the petroleum. The wax precipitated from the slowly cooled petroleum is crystalline and local supercooling of the petroleum is avoided.

T. S. WHEELER.

Distilling oil. L. E. HIRT (U.S.P. 1,559,701, 3,11.25. Appl., 2.3.20).—Distillation takes place in a series of tubes through which the oil flows in thin films. A gaseous medium other than the hydrocarbon being distilled is forced through the tubes to remove mechanically the vaporised constituents of the oil.

H. MOORE.

Apparatus for distilling oil. G. W. WALLACE (U.S.P. 1,561,758, 17.11.25. Appl., 20.5.22).—A still for cracking oil consists of banks of tubes with transverse headers, arranged in a passageway. The products of combustion circulate longitudinally to the tubes, in which endless chains are arranged, travelling around sheaves in the headers, for the removal of carbon. The collected carbon is discharged through an opening in a chamber at the bottom side of one header.

H. MOORE.

Extracting waxes from crude petroleum. E. PETTY, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,559,981, 3.11.25. Appl., 15.9.21).—Wax is extracted in amorphous or crystalline form from paraffin-base crude oils by subjecting them to refrigeration and centrifugal treatment. The wax obtained is distilled to drive off crystalline wax, leaving amorphous wax as a residue.

H. MOORE.

Producing wax-free products from crude petroleum. E. PETTY, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,559,982, 3.11.25. Appl., 11.7.23).—To obtain a wax-free low-pour, bright stock, paraffin-base, crude oil from which the wax distillate has not been removed is subjected to refrigeration and centrifugal treatment. The wax-free oil is distilled, and the residue diluted with a low-boiling petroleum distillate. The blended stock thus formed is subjected to successive treatments with acid and alkali, and the diluent distilled off.

H. MOORE.

Recovery of gasoline etc. H. B. BERNARD, Assr. to SINCLAIR OIL AND GAS CO. (U.S.P. 1,560,137, 3.11.25. Appl., 8.5.24).—Absorbed gasoline is separated from a liquid absorbing medium by distillation. The heated liquid residuum from distillation is passed through heat exchangers in countercurrent with the distilled vapours under a lower pressure than that of the distillation treatment.
H. MOORE.

Gasoline recovery. H. B. BERNARD, Assr. to SINCLAIR OIL and GAS CO. (U.S.P. 1,560,138, 3.11.25. Appl., 27.5.24).—Natural gas, casinghead gas, etc. are treated with a liquid absorbing medium, which is then distilled, and the hot distillate passed through heat exchangers in counter-current with the vapours from the distillation treatment. H. MOORE.

Cracking still for mineral oils. W. L. BAGWILL (U.S.P. 1,560,891, 10.11.25. Appl., 27.12.23).—A number of superposed pipes for combustion gases extend through the still lengthwise from end to end, the uppermost passage being of largest diameter and the others progressively diminishing in diameter. The still is ovate in cross-section, with flat sides and the apex at the bottom, and the heating pipes are so mounted that the distance between the lateral wall of the still and the heating pipes is the same throughout.

Hydrocarbon distillation. H. G. W. KITTREDGE, Assr. to C. A. KITTREDGE (U.S.P. 1,561,169, 10.11.25. Appl., 13.8.19).—The still comprises two reservoirs, the first having an arched top exposed to the heat from the furnace. The lower portion of this reservoir, intended to receive any deposit, is insulated from direct heating by means of brickwork. After passing over the first reservoir the furnace gases pass by means of a fire-tube through a second reservoir surmounted by a column and attached condenser. The liquid level maintained in the second reservoir is such that the fire-tube is completely immersed. Oil is passed through the first reservoir to the second.
T. A. SMITH.

Apparatus for distillation [of oils]. W. M. FRASER (U.S.P. 1,561,428, 10.11.25. Appl., 11.6.23).—A still is provided with an agitator consisting of a continuous band, fitted with scraper plates, and driven by a fluid-motor operated by the pressure of the ingoing oil. T. A. SMITH.

Cracking petroleum. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,561,779, 17.11.25. Appl., 19.10.23).—The oil passes through a coil in which it is subjected to cracking conditions of heat and pressure. Thence a portion of the oil passes to a primary expansion chamber, and from this to a secondary chamber. The remainder of the oil from the coil is introduced into the oil in the secondary chamber to increase conversion therein. H. MOORE.

Treating oils. P. W. PRUTZMAN, Assr. to GEN. PETROLEUM CORP. (U.S.P. 1,561,999, 17.11.25. Appl., 2.9.22).—To purify and partly decolorise oil, a portion of it is mixed with a powdered porous adsorbent material, and the mixture allowed to rest till the air is displaced from the pores of the adsorbent. The remainder of the oil is heated above 176° and added to the mixture. The spent adsorbent is separated mechanically from the decolorised oil. H. MOORE.

Treating [decolorising] oil. P. W. PRUTZMAN and C. J. VON BIBRA, Assrs. to GEN. PETROLEUM CORP. (U.S.P. 1,562,001, 17.11.25. Appl., 2.9.22).—A pulverulent solid decolorising material is prepared in the form of an aqueous mud, air being thus excluded from the pores of the material. The mud is mixed with the oil, and heat applied to drive off water, the solid decolorising agent being thus brought into intimate contact with the oil.
H. MOORE.

Removing acids from oils. P. W. PRUTZMAN and P. D. BARTON, Assrs. to GEN. PETROLEUM CORP. (U.S.P. 1,562,000, 17.11.25. Appl., 2.9.22).—Acid tar is removed from acid-treated petroleum oil by agitating it with a powdered adsorbent which has expended its adsorptive value in removing impurities from tar-free oil and is charged with such oil. H. MOORE.

Purifying mineral oils. J. F. FABER, H. C. HANNA, and M. L. CHAPPELL, Assrs. to STANDARD OIL CO. (U.S.P. 1,562,156, 17.11.75. Appl., 26.1.22). —Petroleum is treated with sulphuric acid, separated from the acid sludge, and neutralised. To prevent the formation of a persistent emulsion on neutralising the treated oil, salts of petroleum carboxylic acids, obtained by extracting the carboxylic acids from crude petroleum oil by means of an aqueous solution of a metal base, are added.

Calorimeter. G. E. SCHOLES (U.S.P. 1,563,662, 1.12.25. Appl., 23.3.25).—See E.P. 236,980; B., 1925, 788.

Treatment of finely-divided coal and production of briquettes. E. EDSER and W. H. BEASLEY, Assrs. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,562,876, 24.11.25. Appl., 29.7.22).—See E.P. 189,220; J., 1923, 86A.

Manufacturing decolorising carbon. J. N. A. SAUER (U.S.P. 1,563,295, 24.11.25. Appl., 27.2.20). —See E.P. 189,148; B., 1924, 364.

Purifying mineral oil products and the like. F. SCHWARZ (E.P. 242,317, 9.5.24).—See G.P. 403,135; B., 1925, 64. (Reference is directed in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 26,198 of 1904, 16,134 of 1901, and 9179 of 1894).

Apparatus for fractionation of petroleum. H. P. STRAUS (U.S.P. 1,562,009, 17.11.25. Appl., 19.6.20).—See E.P. 189,239; J., 1923, 136 A.

Purifying hydrocarbons. A. GREENSPAN (U.S.P. 1,562,425, 17.11.25. Appl., 10.11.22).—See E.P. 203,098; J., 1923, 1061 A.

Process of distillation [of oils]. D. PYZEL, Assr. to SIMPLEX REFINING Co. (U.S.P. 1,562,723, 24.11.25. Appl., 3.11.19. Renewed 11.5.25).—See E.P. 173,907 ; J., 1922, 168 A.

Lubricating compound. A. G. MARSHALL (U.S.P. 1,561,261, 10.11.25. Appl., 17.7.23).—See E.P. 211,978 ; B., 1924, 366.

System of combustion. T. J. J. WASLEY and F. G. SIBILLA (E.P. 242,333, 31.7.24).

Machines for charging and discharging gas retorts and the like. T. F. CANNING and R. G. CLARK (E.P. 242,369, 16.8 and 22.9.24).

Manufacture of briquettes from coke smalls or the smalls of other lean fuels. L. WEBER (E.P. 243,129, 13.10.24).

Safety device for gas and air mixing plants and other industrial gas plants. SELAS A.-G. (E.P. 235,164, 5.5.25. Conv., 4.6.24).

Production of air-gas mixtures for combustion. H. SALAU, and BADER UND SALAU (E.P. 243,222, 14.4.25).

Transmitting heat from one medium to another. (U.S.P. 1,560,271).—See I.

Lead tetra-alkyl. (U.S.P. 1,559,405).—See XX.

III.—TAR AND TAR PRODUCTS.

Blue oil of lignite tar. J. HERZENBERG and S. RUHEMANN (Ber., 1925, 58, 2249—2262).—The neutral oil of lignite tar contains a dark blue component which can be concentrated by treatment of the oil with aqueous hydroferrocyanic acid followed by decomposition of the pale yellow ferrocyanide with dilute sodium hydroxide and fractional distillation of the liberated oil, but the fractions are highly complex mixtures from which the blue component cannot be isolated. Its sensitiveness towards air and mild oxidising agents and also towards reducing agents is remarkable. Further examination of it is greatly facilitated by the spectroscopic proof of its identity with the blue hydrocarbon azulene, derived from many essential oils and isolated by Sherndal (J., 1915, 150, 730) as picrate. Azulene is readily prepared by the catalytic dehydrogenation of gurjunene at 400—410° by nickel supported on aluminium oxide ; the distillate is either fractionated and the fractions of highest b.p. treated with picric acid or the total distillate is treated with hydroferrocyanic acid and the oil recovered from the ferrocyanide is transformed into the picrate. Azulene probably owes its presence in lignite tar oil to the dehydrogenation of sesquiterpenes of the gurjunene type formed by pyrogenic decomposition of the resinous components of bitumen. In confirmation, it is found that a nearly colourless, neutral fraction, b.p. 126—133°/13 mm. gives an intensely blue distillate when dehydrogenated over a nickel catalyst at 340—350°, whereas fractions of higher b.p. appear to contain only traces of sesquiterpenes capable of forming azulene. The possibility that finely-divided carbon is the catalyst during the production of the tar is suggested by the observation that a fraction of gurjun balsam oil, b.p. 127·3—128°/13 mm., loses hydrogen and affords a dark blue distillate when passed over activated charcoal at 410—420°. The production of blue oils by the action of sulphuric acid and acetic anhydride on bicyclic sesquiterpenes, such as cadinene, and terpenes has frequently been reported. Highly purified samples of *d*-sylvestrene, cadinene, and caryophyllene, however, give colourless or almost colourless distillates when subjected to catalytic decomposition. The latter process, therefore, affords a very sensitive method of testing the freedom of terpenes and sesquiterpenes from azulene-forming terpenes of the gurjunene type. H. WREN.

Process for working up tar-oil fractions with a high creosote content. E. KÁRPÁTI (Chem. Runds. Mitteleuropa u. Balkan, 1925, 2, 229—230 ; Chem. Zentr., 1925, 96, II., 2036).—Tar oils are worked up to neutral oils, by converting the phenolic constituents into non-volatile condensation products by treatment with concentrated sulphuric acid or formaldehyde. In using concentrated sulphuric acid, the oil is subsequently separated by distillation *in vacuo*, and the residues are used for tanning. Formaldehyde yields condensation products resembling bakelite, from which the neutral oil is separated by distillation in a current of superheated steam. L. A. COLES.

PATENTS.

Removing phenol and/or its homologues from liquids such as waste waters or the like. P. PREISS (E.P. 241,682, 19.9.24).—The liquid from which it is desired to extract phenols is rendered neutral or slightly acid, and is sprayed down a tower in which it meets an ascending current of vapour of benzol, benzine, or trichloroethylene. The last-named is most suitable. The two liquids are separated at the bottom of the tower and the trichloroethylene with the extracted phenols is returned to a still. The phenols will gradually accumulate, and are removed from time to time. C. IRWIN.

Separating phenols from crude oils. V. L. OIL PROCESSES, LTD., O. D. LUCAS, and E. L. LOMAX (E.P. 242,092, 24.11.24).—Sodium phenoxide solution, obtained by treating oil containing phenols with sodium hydroxide solution of at least 20% strength, is used for extracting phenols from further quantities of the oil, yielding a solution containing sodium phenoxide and free phenols. The phenols are precipitated by dilution with water, and are removed, and the residual solution, after concentration to suitable strength, is used again. If the sodium hydroxide solution is at or above 30% strength, part of the neutral oil dissolves together with the phenols, but can be removed by steam distillation. L. A. COLES.

Distillation of benzol wash-oil (G.P. 417,340).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Proposed system of tests for the fastness of dyestuffs on wool. H. R. HIRST (J. Soc. Dyers and Col., 1925, **41**, 347—354).—The methods given are based on actual experience of the use of the dyestuffs in the cloth manufacturing and dyeing trades and are published with the view of establishing standard methods for testing, depending on both manufacturers' and wearers' requirements. The results of the tests are estimated by comparison with dyed patterns retained as standards and the changes are judged by inspection, the results so obtained being considered of more value than those obtained with scientific instruments. The usual five degrees of fastness have been adopted and the standards are those recommended by the " German Commission on Fastness of Dyes," 1914. Fastness to light.—Dyes for outside purposes should be directly exposed to sunlight and weather, whilst those for indoor use should be exposed under glass. As standards for light tests, the following dyes (in ascending order of fastness) have been selected: 0.—4% Indigo Carmine X (B.D.C.). I.—1·5% Ponceau R.G. (B.D.C.). II.—2·75% Azo-Rubine (Sand.). III.—4·5% Kiton Red S. (C.A.C.). IV.—2·5% Chlorazol Fast Red F.G. (B.D.C.). V.—Indigo (full shade) or Naphthol Green B. (C.). Special light exposures should be made where the patterns are exposed to sea-spray or to the air in industrial areas. Fastness to washing.—In this as in most of the subsequent tests the dyed worsted yarn is plaited with white wool, cotton, mercerised cotton, and silk. The test consists in giving a mild scour at 40° and then a strong scour at 80° (each for 15 mins.) with a solution containing 1% of soap and 0·5% of soda ash. Fastness to cold water is determined by placing the dyed yarn in distilled water at 20° for 12 hrs. (there is a distinct difference in results when using tap water). Fastness to rubbing is tested after the dyed material has been well washed without further cleansing. If the dye is for loose material (wool, tops or yarn) the test may be made after scouring and milling. White cotton cloth is rubbed vigorously ten times backwards and forwards over a 10 cm. length with the dyed pattern and the effect noted both for dry and moist fabrics. Fastness to hot ironing.—The pattern (as dyed, if for piece dyeing, or scoured if for loose wool) is pressed for 10 secs. with a hot iron of such a temperature that it does not scorch white flannel. Fastness to stoving.—The dyed yarn is soaped with a 0·2% solution, hydro-extracted, and then left for 12 hrs. under a bell jar in which sulphur is burned, a standard dye being placed alongside. Fastness to perspiration.—The wetted-out pattern is placed in a 10% solution of salt containing 5 c.c. of a 30% solution of ammonium acetate, and incubated for 24 hrs. at 40°. Dyes for saddle cloth materials should be fast to alkali. Fastness to alkali (street-dust fastness) is determined by spotting the dyed material with a mixture of 10 g. of quicklime and 6 c.c. of ammonia (*d* 0·880) in 1000 c.c. of water. After drying the cloth is brushed and classed according to its appearance. Fastness to acid.—The dyed yarn is treated with a 0·25% solution of sodium bisulphate (70 times the weight of the cloth) for 1½ hrs. at 90—92°. Fastness to peroxide bleaching.—The testing bath consists of 100 pts. of distilled water, 20 pts. of hydrogen peroxide (10—12% volume), made just alkaline and maintained alkaline with ammonia. The material is entered at 45°—50° and left for 12 hrs. Fastness to milling.—The knitted yarn is treated (i) with 40 times its weight of a 2% soap solution, well worked, and kept in the liquor for 2 hrs. at 30°, (ii) in the same liquor with 0·5% of soda ash added, at 50°. The tests should be confirmed by actual milling. Fastness to carbonising.—The sample is steeped in dilute sulphuric acid (*d* 1·035) for ½ hr., squeezed so as to leave 100% of the solution, and heated for 1 hr. at 80° when the cotton should be carbonised. Fastness to potting.—Patterns previously milled are heated with 6 times their weight of water at 90° for 2 hrs. Decatising.—The test should be made in a practical manner on some approved machine. Fastness to sea-water.—The dyed yarn is kept for 24 hrs. in 40 times its weight of sea-water and dried without washing. Methods of dyeing the type samples on wool, and a list of the standard dyestuffs required are given. It is emphasised that where dyed silk and cotton are blended with wool for effect purposes they have to stand all the requirements for finishing and hence the tests for fastness should be identical in all the industries concerned. A. COULTHARD.

Benzidine derivatives of thiocarbamide [and azo dyestuffs therefrom]. L. PINTO (Compt. rend., 1925, **181**, 788—790).—Monoacetylbenzidine when warmed with carbon disulphide in alcoholic solution yields diacetyldibenzidinethiocarbamide, m.p. above 360°, which is hydrolysed by heating with 10% hydrochloric acid at 125—130°, giving dibenzidinethiocarbamide. The latter when diazotised couples with azo components to give direct cotton dyestuffs, *e.g.*, with aminonaphtholdisulphonic acid (H acid) to a blue, with sulphophenylmethylpyrazalone to a yellow, and with β-naphthol to a red dye.
L. F. HEWITT.

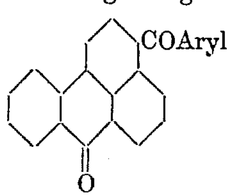
PATENTS.

Manufacture of new [azo] dyestuffs. SOC. OF CHEM. IND. IN BASLE (E.P. 220,303, 1.8.24. Conv., 11.8.23).—*Dyestuffs* giving on cellulose acetate, in an acetic acid bath, yellow to blue and brown shades, of good fastness to washing and rubbing and excellent fastness to light, are obtained by coupling unsulphonated nitro-diazo-compounds of the benzene series (except those having a hydroxy-group ortho to the diazo-group) with monosulphonic acids capable of being coupled, derived from *N*-aryl-, *N*-alkylaryl-, *N*-aralkyl-, and *N*-alkylaralkyl-derivatives of aniline or its homologues or substitution products (other than the *N*-alkylaralkyl-derivatives of metanilic and *m*-toluidinesulphonic acids), or by coupling monosulphonic acids of the same nitro-diazo-compounds (other than the *m*-nitroanilinesulphonic acid of E.P. 20,313 of 1895; cf. J., 1896, 708) with the above amines non-sulphonated. For example, 183 pts. of 2 : 4-dinitroaniline are diazotised with the

calculated quantity of nitrosyl sulphate in concentrated sulphuric acid and the mixture is poured on ice. The product is added, while stirring, to a solution of 313 pts. of sodium ethylbenzylaniline-sulphonate. The dyestuff (which separates rapidly), after being filtered off and dried, is a bronze powder, giving fast bluish-red shades. A table showing thirteen examples is given. A. COULTHARD.

Production of azo dyestuffs insoluble in water. G. KALISCHER AND K. KELLER, Assrs. to L. CASELLA U. Co (U.S.P. 1,558,890, 27.10.25. Appl., 24.3.25).—A diazo-compound is combined with a derivative of an arylide of 2:3-hydroxynaphthoic acid with the general formula $C_{10}H_6(OH)\cdot CO\cdot NH\cdot R\cdot X\cdot R\cdot NH\cdot CO\cdot C_{10}H_6\cdot OH$; R is an aryl residue and X an azo- or azoxy-group. Coupling occurs in both the 1-positions to give *dyestuffs* which yield fast red, claret, blue, and black shades on cotton. These shades are best produced in the fibre by soaking cotton in an alkaline solution of the arylide and then immersing in a bath of the diazo-compound. The azo- or azoxy-arylides are prepared by condensing the chloride of 2:3-hydroxynaphthoic acid or its *O*-acyl derivatives with aromatic diamino-azoxy- or -azo-compounds and saponifying when using an *O*-acyl derivative. Alternatively the nitro-arylides of 2:3-hydroxynaphthoic acid are reduced in alkaline solution. For example the *m*-nitro-*o*-toluidide of 2:3-hydroxynaphthoic acid is reduced in alkaline solution to the corresponding azoxy-compound, which is dissolved in alkali to give a bath in which cotton is soaked. On immersing the soaked cotton in a bath of diazotised *m*-nitro-*p*-toluidine a fast bluish Turkey-red shade is obtained. The azoxy reduction product of the *m*-nitroanilide of 2:3-hydroxynaphthoic acid coupled with diazotised *m*-nitraniline gives yellow-orange shades, with diazotised *p*-nitro-*o*-toluidide blue-red, with diazotised aminoazotoluene claret, and with tetrazotised *o.o'*-dianisidine blue shades. T. S. WHEELER.

Manufacture of condensation products [dyestuffs] of the anthraquinone series. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 220,304, 1.8.24. Conv., 8.8.23. Addn. to 205,502).—1-Acylbenzanthrones having the general formula,



obtained by the successive conversion of the nitro-group in 1-nitrobenzanthrone or its substitution products, into the amino-group, the cyano-group, the carboxyl group, the carboxylic acid chloride group, and finally, by treatment with an aromatic hydrocarbon in the presence of aluminium chloride, into the acyl group, are converted into products identical with those described in the chief patent (cf. B., 1924, 627) by treatment with aluminium chloride at high temperatures. For example, 1-cyanobenzanthrone, m.p. 335—341°, is hydrolysed with a mixture of 3 pts. by vol. of concentrated sulphuric acid and 2 pts. of water, yielding benzanthrone-1-carboxylic acid, m.p. 350—355°. After conversion to the acid chloride, the product is stirred at the ordinary temperature and then heated on the water bath with excess of benzene and aluminium chloride, yielding 1-benzoylbenzanthrone, m.p. 194—195°, which on heating with 15 pts. of aluminium chloride at 180° for 2 days, yields a dyestuff of m.p. 385°, identical with that obtained as described in example 1 of the chief patent. L. A. COLES.

Manufacture of vat [anthraquinone] colouring matters. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 242,837, 18.2.25. Addn. to 204,249).—1-Halogen-2-aminoanthraquinones or their derivatives are treated with aromatic compounds containing at least two substituents with reactive carbon atoms, such as carbonyl chloride groups, or di- or tri-halogenmethyl groups, or, alternatively, they may be treated with oxalyl chloride or adipyl chloride, or with other aliphatic dicarboxylic acid chlorides, and the products are treated in the presence or absence of condensing agents, with sulphides, polysulphides, xanthates, or other compounds capable of yielding sulphur. For example a mixture of 10 pts. of the product obtained by the action of 1 mol. of terephthaloyl chloride on 2 mols. of 1-chloro-2-aminoanthraquinone, 40 pts. of crystalline sodium sulphide, 10 pts. of sulphur, 100 pts. of water, and 100 pts. of 96% alcohol, is stirred for 3 hrs. at 180—200° under pressure. After distilling off the alcohol and diluting the residue with water, the dyestuff is filtered off, washed, and purified if necessary by treatment with sodium hypochlorite solution. The product is identical with that obtained from 1-mercapto-2-aminoanthraquinone and terephthaloyl chloride by the process described in the chief patent (*cf.* U.S.P. 1,459,536; J., 1923, 881A). L. A. COLES.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Polysaccharides XXXIII. Enzymic cleavage of artificial silk and natural cellulose. P. KARRER, P. SCHUBERT, and W. WEHRLI (Helv. chim. Acta, 1925, 8, 797—810; cf. B., 1925, 441, 585).—The way in which cellulose is precipitated exerts a profound influence on the rate of enzymic hydrolysis; *e.g.*, viscose cellulose suffers 57·5% decomposition in the same time as cuprammonium cellulose undergoes 7·8% hydrolysis. The differences in resistance to the action of cellulase are ascribed to differing micellar structures. When the dilution of the enzyme solution is increased in a geometrical ratio the amount of decomposition decreases in an arithmetical ratio. Hence it is necessary for practical application of the enzymic cleavage to maintain the concentration of enzyme as high as possible. The hydrolysis is at first approximately

unimolecular. The effect of doubling the enzyme content is to increase about 1½ times the amount of sugar-formation. Different brands of commercial viscose artificial silk show marked differences in their behaviour towards cellulase. Further, the defective parts known as "milky" spots which are sometimes found in viscose silk tissues are hydrolysed by the enzyme much more readily than the undamaged portion. The enzymic hydrolysis of natural cellulose by snail cellulase proceeds only very slowly (20% in 32 days in one example). The efficiency of the enzyme falls from 17·2% at 35° to 15% at 50° and 3·2% at 60°. R. A. MORTON.

Production of cellulose with the aid of chlorine. W. SCHACHT (Papier-Fabr., 1925, **23**, 759—762).—Sodium hydroxide and chlorine are used for the production of cellulose from vegetable materials, such as grasses, which contain but little lignin. The use of chlorine has the disadvantage that hydrochloric acid is formed, which may damage the cellulose. When gaseous chlorine is used the dissolution of the hydrochloric acid causes a rise of temperature, and the process is less under control than with aqueous chlorine. It is preferable to remove most of the lignin by treatment with hypochlorite ; only a minimum quantity of free chlorine is then necessary. A vigorous chlorine treatment also leads to a considerable loss in weight. Paper made from esparto cellulose produced by a severe chlorination has the properties of an ordinary chemical pulp paper, rather than those of a good esparto paper. The use of sodium hydroxide and chlorine is economical if these can be obtained by the electrolysis of sodium chloride, using cheap water power. A. GEAKE.

Particle size of wood cellulose. D. KRÜGER (Papier-Fabr., 1925, **23**, 767—768).—The particle size of cellulose can be measured by nitrating carefully, dissolving the nitrate in acetone, and measuring the rate of diffusion. The particles of a sulphate-cellulose had a diameter of $1·4\times10^{-6}$ cm., those of two samples of sulphite-cellulose 2·2 and $2·4\times10^{-6}$ cm., and of cotton $3·4\times10^{-6}$ cm. Cotton has thus appreciably larger particles than the other celluloses, accounting for the lesser solubility and greater viscosity of its esters. The ripening of alkali-cellulose in viscose manufacture results in a reduction of the particle size, as do also the bleaching of sulphite-cellulose with hypochlorite, and the beating of hemp-cellulose. The viscosity of nitrocellulose solutions falls with diminishing particle size, and it may, therefore, be possible to determine the particle size and ripeness of celluloses by measuring the viscosity of the nitrate, prepared under standard conditions. A. GEAKE.

Cellulose esters of higher fatty acids. G. KITA, T. MAZUME, I. SAKURADA, and S. NAKAJIMA (Cellulose Ind., Tokyo, 1925, **1**, 227—232).—Only a slight degree of esterification is obtained by heating cellulose with palmitic anhydride in the presence of pyridine and chloroform. On the other hand, when cellulose is heated with palmityl chloride and pyridine, preferably diluted with benzene, progressive formation of esters takes place, with the ultimate production of cellulose tripalmitate soluble in benzene or ether. The esters are purified by washing the product with alcohol and then extracting with ether or benzene. The monopalmitate is still fibrous and only slightly swollen ; the higher palmitates while retaining a fibrous structure are very much swollen and completely deformed. The tripalmitate from hydrocellulose is completely soluble in ether, that from normal cellulose only partly soluble. The dipalmitate from hydrocellulose is soluble in benzene, but that from normal cellulose only partly. The monopalmitate is insoluble in benzene but becomes transparent in that liquid. The solutions from ether or benzene deposit elastic films. The melting points, ranging from 180° to 220°, are lower in the esters from hydrocellulose than in those from normal cellulose. In the analysis by saponification, the addition of benzene to the alcoholic potassium hydroxide promotes the reaction. J. F. BRIGGS.

Properties of viscose. G. KITA, R. TOMIHISA, and H. ICHIKAWA (Cellulose Ind., Tokyo, 1925, **1**, 193—200).—The quantity of sodium combined as xanthate increases with the concentration of the alkali used for dissolving the viscose ; it reaches a maximum after the viscose has attained a certain degree of ripening. The viscosity of viscose dissolved in strong alkali decreases during ripening and that of viscose dissolved in weak alkali rises at first and decreases only at a later stage. High alkalinity of the viscose favours the disappearance of the fibrous structure, whilst viscose dissolved in water only always shows fibres. Viscose dissolved in strong alkali gives stronger films ; the strength of the film tends to increase with the percentage of combined sodium. In these circumstances the strength of the film tends to increase to a maximum, despite the fact that the viscosity gradually falls. The strength of the film from viscose dissolved in water only is poor, although the viscosity is high. Reduction of viscosity by long ripening of the alkali-cellulose or by mercerising with concentrated sodium hydroxide does not necessarily decrease the strength of the film. Xanthates prepared with small proportions of carbon disulphide always give low values. J. F. BRIGGS.

Composition of viscose. G. KITA, R. TOMIHASA, and S. IWASAKI (Cellulose Ind., Tokyo, 1925, **1**, 129—134).—The usual methods for the determination of combined sodium and combined sulphur in viscose are considered to be defective. According to the preferred procedure, 2 g. of a 6% viscose solution are acidified with 5 c.c. of 0·5- or 0·25N-acetic acid, and the xanthate is salted out and washed with a neutral saturated solution of sodium chloride. The xanthate is decomposed with 15 c.c. of 0·02N-hydrochloric acid, allowed to stand for 4 hrs., the precipitate is filtered off and washed, and the excess of acid in the filtrate is titrated. The combined sulphur is determined by Carius' method in the xanthate similarly purified as above. The combined alkali found by this method never exceeds 0·34 mol. per mol. of

$C_6H_{10}O_5$. It is lower, the higher the concentration of the caustic soda used for mercerising, the longer the ripening of the alkali-cellulose, and the smaller the quantity of carbon disulphide used. When the viscose is matured for a week the proportion of combined sodium does not change very much but increases to a maximum and then decreases again. The combined sulphur corresponds with the combined sodium, but is always about 10% higher than the equivalent quantity of sodium. The viscosity of the viscose is lower, the lower the proportion of combined sodium and sulphur. J. F. BRIGGS.

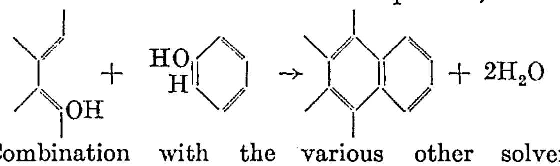
Cellulose fibre and tissue paper as materials for celluloid. K. ATSUKI (Cellulose Ind., Tokyo, 1925, 1, 3—13).—Normal cotton cellulose, well purified, is the only acceptable raw material for celluloid; it may be in the form of cotton wool or tissue paper. Other celluloses, such as those from wood, straw, or bast fibres, either contain too much non-resistant cellulose or are chemically modified by the processes of digestion. Old cotton rags are similarly unsuitable for the manufacture of tissue papers for celluloid. Loose cotton wool prepared from spinning wastes gives nitrocellulose of the highest viscosity, but unless the solvent and mechanical means for dispersion are exceptionally well adapted the celluloid is apt to be brittle. Such celluloid is best suited for the manufacture of heavy, rigid articles. Tissue paper made from sound cotton material has the advantage over cotton wool of greater cleanliness, and the properties of the celluloid may be controlled to a great extent by the treatment of the pulp. Wet beating lowers the viscosity of the nitrocellulose and gives a celluloid which is soft and plastic. The correct beating should be sharp and free, to afford uniform nitration, with a slight amount of developed hydration according to the plasticity desired. The best thickness is between 0·04 and 0·07 mm.; thick papers are not uniformly nitrated. The structure of the sheet should be as open as possible provided it is uniform. The ratio of substance in g. per sq. m. to thickness in mm. should be less than 600. The chemical purification of the raw material must be carefully controlled. The use of coloured rags for a bleached paper is condemned. Colour due to traces of iron is not very harmful, as it is removed by the acids; colour due to organic matters must be eliminated by careful bleaching. A standard colour for celluloid tissue paper may be taken as 0·05 by the Lovibond tintometer.
J. F. BRIGGS.

Stability of nitrocelluloses. J. DUCLAUX (Rev. gén. Colloid., 1925, 3, 257—263).—The stability of nitrocellulose is determined not only by the chemical character of the nitrocellulose, but also, though in a less marked degree, by its physical constitution or ultramicroscopic structure; physical structure varies in the case of films and the like with the method of production, as evidenced by differences in optical properties, mechanical properties, and apparent density of films of the same thickness made from the same sample of nitrocellulose but with the aid of different dispersion media. For instance, the higher-boiling solvents such as *iso*butyl formate or mesityl oxide give with the same sample of nitrocellulose films of higher stability than do the low-boiling solvents or solvent mixtures such as acetone, ether-alcohol, or the like. Moreover with high-boiling solvents slight variations in the conditions obtaining during the formation of the film have no appreciable effect on the stability, whereas with low-boiling solvents concordant results are difficult to obtain. Quite small quantities of high-boiling solvents in admixture with low-boiling solvents are effective in giving films of constant physical structure and hence constant stability, provided that, immediately prior to solidification, only the high-boiling solvent remains in association with the nitrocellulose. There appears to be no definite relationship between stability and apparent density. It is conceivable that the stabilising action of amyl alcohol and diphenylamine is due in part to a modification of the physical structure of the nitrocellulose.
D. J. NORMAN.

Effect of chemicals on paper-making properties of cellulose. R. SIEBER (Papier-Fabr., 1925, 23, 765—767).—By treatment of a sulphate-cellulose with 1% sulphuric acid for 3 hrs. at 50° the strength of the paper made from it was reduced 13%, but the beating properties were not affected; treatment for 2 days with 2% lactic acid at 30° or for 1 day with 5% sodium hydroxide at the ordinary temperature produced no effect. Boiling calcium or barium hydroxide injured both the strength and the beating properties, but the latter were partially restored by subsequent acid treatment. Beating was less efficient when 10% of sodium sulphate was added to the water and when the material was previously dried for 14 days at 100°. A. GEAKE.

Solubility of lignin in phenols. A. HILLMER (Cellulosechem., 1925, 6, 169—187; cf. G.P. 412,235, B., 1925, 707).—The solubility of lignin in a large number of organic compounds has been examined qualitatively, 1 g. of lignin being taken to 50 g. of solvent. In many cases hydrochloric acid lignin (residue from hydrolysis of wood with cold 42% hydrochloric acid) was used, it being found that its solubility was similar to that of lignin itself. Among phenolic compounds the best solvents are those containing at least two hydroxyl groups, or a carboxyl group or a nitro-halogen, or aldehyde group in the *m*- or *p*-position to a hydroxyl group. Dissolution is greatly assisted by catalysts; chlorine and bromine are as effective in this respect as the strong mineral acids and have the advantage of not hydrolysing cellulose. Other good solvents for lignin are the stronger organic acids, such as halogen-acetic acids, succinic acid, maleic acid, *o*-nitrophenylacetic acid, mandelic acid, halogen- and nitro-benzoic acids, and benzenesulphonic acid; *p*-nitroaniline and *p*-toluenesulphonyl chloride are also good solvents. Quantitative experiments were made with *p*-chlorophenol. The amount of lignin dissolved depends greatly on the temperature and the duration of the digestion; the dissolution of

accompanying cellulose is hindered by the addition of bases, *e.g.*, 5% of pyridine. In the presence of catalysts good separation of lignin and cellulose is obtained without the addition of bases; for this purpose iodine was used, this being a less active catalyst than chlorine or bromine. By treatment for 30 min. at 80° with creosote and 0·2% of iodine, 98% of an acid lignin is dissolved, whereas cotton wool remains unattacked. The dissolution of lignin in phenols is not a simple process of solution, but chemical combination takes place; it is suggested that lignin contains a reactive carbonyl group, which condenses in the enolic form with phenols,



Combination with the various other solvents mentioned can be represented similarly. Phenol-lignins are obtained from the phenolic solutions by concentrating in a vacuum to a syrup and pouring into ether. The substance thus precipitated contains the particular phenol used as a solvent, as is proved by the presence of nitrogen and chlorine, respectively, in the phenol-lignins from *o*-nitrophenol and *p*-chlorophenol. The course of the reaction cannot be deduced from the composition of the primary phenol-lignins, prepared from natural lignin, as the composition of this latter is unknown. From an acid lignin having the composition $C_{37·5}H_{33·1}O_{9·4}(OMe)_4$ a secondary phenol-lignin was prepared, the analysis of which corresponded to the addition of 1 mol. of phenol and the loss of 2 mols. of water and 1 of methyl alcohol. The phenol-lignins are amorphous substances readily soluble in acetic acid, alcohols, ethyl acetate, acetone, pyridine, quinoline, aniline, phenols, and dilute alkali hydroxides; they are insoluble in water, ether, chloroform, carbon tetrachloride, carbon disulphide, petroleum, trichloroethylene, benzene, toluene, turpentine oil, aqueous ammonia, sodium carbonate solution, and dilute mineral acids. A. GEAKE.

Resin sizing of paper. R. LORENZ (Ver. Zellstoff- u. Papier-Chem., Hauptversamml., 1924, 78—86; cf. B., 1925, 586, 799).—For the prevention of premature coagulation of the resin size when mixed with hard water in the beater, it is not advantageous to work with highly concentrated emulsions and to restrict the quantity of water in the pulp. The tendency to spontaneous coagulation of a colloidal solution increases with its concentration. Flocculation under the influence of hard water is not governed by the quantity of the hard water but by the concentration of the hardening constituents. The system is most stable at high dilutions, and the colloidal flocculation of the resin by the hydroxide of the earthy constituents of the water is avoided by increasing the dilution of the hydroxyl ions to a point where these earthy hydroxides are not precipitated. The most effective means for preventing flocculation of the resin size emulsions by hard water is the addition of a protective colloid, provided it is not so powerful as to prevent flocculation at a later stage when the alum is mixed with the pulp. When the requisite quantity of concentrated aluminium sulphate is added to the sized pulp in the beater, three different flocculating influences may come into play. The mixture of pulp and size being definitely alkaline, slight chemical precipitation of aluminium hydroxide and some resin takes place in the first stage. The colloidal combination between aluminium hydroxide and resin in virtue of their opposite electrical charges only induces flocculation when the ratio of aluminium hydroxide to resin is about 1:25. Thus, in the second stage, as the aluminium sulphate becomes mixed and diluted, a colloidal flocculation of this order spreads like a wave through the stuff only where the two reagents aluminium hydroxide and resin are in contact in the correct proportions; this kind of coagulation does not occur when the resin is much diluted. The major influence of flocculation is undoubtedly that of the very powerful tervalent aluminium ion which acts steadily throughout without restrictions of conditions. Experiments with the Plauson colloid mill have indicated that effective sizing could be produced by mechanical means alone, by grinding the resin with the pulp to a sufficiently fine state of subdivision.
J. F. BRIGGS.

Methods of distinguishing sulphite- and soda-celluloses in paper. KORN (Papier-Fabr., 1925, 23, 781).—Lofton and Merritt's method for determining the proportions of sulphite- and soda-celluloses in mixtures (J., 1921, 343 A) gives too high results for the former when this is present in small amounts. Correct results are obtained by Wisbar's modification (J., 1923, 711 A). The method of Alexander (cf. Cameron, Paper, 1924, 33, 138) gives a clear distinction in the majority of cases, but soda-celluloses from many coniferous woods give the same red colour as is given by sulphite-celluloses; the test is, therefore, valuable only when the blue colour, yielded by most soda-celluloses, is obtained. A. GEAKE.

Stiffness of paper. H. SCHULZ and W. EWALD (Papier-Fabr., 1925, 23, 768—770).—The stiffness of paper is determined by clamping horizontally a strip 20 mm. wide with a free length of 80 mm.; a weight is rested on the paper and moved along until the strip bends at the clamp. The distance of the weight from the clamp is a measure of the stiffness of the paper. A. GEAKE.

PATENTS.

Protecting furs, wool, and similar materials from attack by moth. E. NAEFE (G.P. 416,706, 9.9.24).—The articles are impregnated with benzene containing in solution antimony soaps, prepared, *e.g.*, by heating antimony pentoxide with concentrated potassium hydroxide solution and adding an excess of oleic acid or other saponifiable fat.
L. A. COLES.

Manufacture of cellulose compounds [thiourethane derivatives]. L. LILIENFELD (E.P.

241,149, 20.11.24. Conv., 11.10.24; *cf.* B., 1925, 955,985).—An ester of an inorganic acid is caused to act upon a thiourethane or *N*-substituted aryl- or alkyl-thiourethane of cellulose (E.P. 231,801; B., 1925, 955) in the presence of an alkali. For example, 100 pts. of a phenyl- or tolyl-thiourethane of cellulose are dissolved in 4900 pts. of 10% sodium hydroxide solution and mixed with 600—1000 pts. of ethyl sulphate, the mixture being agitated. The precipitated product after keeping in the presence of the liquor for several days is separated and washed until free from alkali, then treated with dilute sulphuric acid, thoroughly washed, and dried. The reaction may be performed by the action of ethyl chloride at a low temperature in an autoclave on the cellulose arylthiourethane in the presence of sodium hydroxide, part of which is in the solid state. The products may be regarded as ethyl esters of aryliminothiocarboxylic esters of partially etherified cellulose. They are soluble in most volatile organic solvents including a mixture of alcohol and benzene and in many high-boiling plastifiers. J. F. BRIGGS.

Production of cellulose compounds [ether-esters]. COURTAULDS, LTD., W. H. GLOVER, and E. VAN WEYENBERGH (E.P. 241,679, 15.9.24).—A cellulose ether, containing, say, 26·3% C_2H_5, soluble in cold water and insoluble in 8% sodium hydroxide solution, is heated at 70—90° for 5 hrs. with glacial acetic acid. The product gives a clear solution and when separated by precipitation, it is no longer affected by cold water and is readily soluble in benzene, alcohol, and other organic solvents. Analogous products may be obtained by heating a cellulose ether with formic or propionic acid. The esterification may alternatively be performed at a lower temperature by the action of a lower fatty acid in the presence of a catalyst, *e.g.*, sulphuric acid. J. F. BRIGGS.

Cellulose acetate composition. S. J. CARROLL, Assr. to EASTMAN KODAK CO. (U.S.P. 1,560,542, 10.11.25. Appl., 28.7.24).—The composition is a solution of colloidised cellulose acetate, soluble in acetone, and cresyl *p*-toluenesulphonate. A. GEAKE.

Treating cellulose acetate reaction mixtures. E. S. FARROW, JUN., Assr. to EASTMAN KODAK CO. (U.S.P. 1,560,554, 10.11.25. Appl., 4.8.24).—Cellulose acetate is precipitated from viscous reaction mixtures in a highly divided form by agitating with blades and adding water. A. GEAKE.

Manufacturing cellulose acetate. A. F. SULZER, Assr. to EASTMAN KODAK CO. (U.S.P. 1,560,620, 10.11.25. Appl., 28.1.25).—By heating in a vacuum, acetic acid is removed from the reaction mixture at a temperature below that at which cellulose acetate is degraded. A. GEAKE.

Spinning artificial silk etc. H. P. BASSETT and T. F. BANIGAN, Assrs. to MEIGS, BASSETT and SLAUGHTER, Inc. (U.S.P. 1,560,965, 10.11.25. Appl., 17.5.23).—A solution of cellulose acetonitrate in acetone, issuing from a spinneret, is passed through a shallow body of acetone and water, and the thread thus formed is passed through an appreciable depth of air. A. GEAKE.

Manufacture of artificial silk from nitrocellulose. E. BINDSCHEDLER, Assr. to TUBIZE ARTIFICIAL SILK CO. (U.S.P. 1,562,076, 17.11.25. Appl., 31.5.24).—A solution of nitrocellulose hydrate in alcohol and ether is forced through orifices into a mixture of glycerin and an aliphatic monohydric alcohol, and is drawn through this mixture at a greater speed than through the orifices. A. GEAKE.

Manufacture of viscose artificial silk. F. KÜTTNER (G.P. 416,210, 17.6.23).—Freshly formed viscose filaments (coagulated by means of acid salt solutions) are steamed while being wound from the centrifuge on to bobbins. A. J. HALL.

After-treatment of artificial fibres and other products prepared from viscose or other cellulose compounds. H. VOSS (G.P. 416,557, 7.11.22).—After-treatment is effected by means of solutions containing sulphite-cellulose waste lye and a mineral acid, such as sulphuric acid. A. J. HALL.

Pulp process. Preparation of wood for pulp manufacture. Wood-pulp material. J. H. WALLACE, Assr. to PINE WASTE PRODUCTS, INC. (U.S.P. 1,560,446—8, 3.11.25. Appl., 25.8.20).—(A) Resinous wood is extracted by means of a volatile solvent so long as the extraction proceeds rapidly, and the extracted wood is then converted into a pulp by treatment with hot alkaline lyes, the residual non-volatile and solid resinous matters in the spent alkaline lyes being recovered subsequently. (B) Porous long-fibred wood chips suitable for the manufacture of wood pulp and containing less than the normal amount of resinous substances (uniformly distributed) are prepared by converting resinous coniferous wood into chips and subjecting these to solvent extraction only so long as the extraction proceeds rapidly, so that the resulting chips contain a substantial amount of substances capable of extraction by the solvent. (C) Claim is made for wood chips prepared by the process described in (B). A. J. HALL.

Process for making paper pulp. B. T. MCBAIN, J. E. ALEXANDER, and G. GENBERG, Assrs. to NEKOOSA-EDWARDS PAPER CO. (U.S.P. 1,560,591, 10.11.25. Appl., 2.9.22).—Resinous wood is treated by the usual sulphate process, and the pulp thus obtained is treated with an acid sulphite liquor. A. GEAKE.

Quick-cook sulphite process. P. C. ULMEN, H. H. BECKER, and R. T. MANN (U.S.P. 1,560,881, 10.11.25. Appl., 14.4.24).—When the digestion process is complete, cold water is injected while the spent liquor is being drained off, in order to lower quickly the temperature and pressure within the digester, and the pulp is then washed out.

Control of bisulphite-pulp cooking. J. E. FLEURY (U.S.P. 1,562,217, 17.11.25. Appl., 14.5.24).—Samples of liquor from a digester are treated with

an alkali hydroxide and an alcohol, filtered, and compared in a colorimeter with standards.

A. Geake.

Recovering sodium acid sulphite from waste liquors of sodium acid sulphite pulp process. J. Beveridge, Assr. to J. B. Beveridge (U.S.P. 1,560,649, 10.11.25. Appl., 12.12.24).—Waste liquor from the sodium acid sulphite wood pulp process is drained from the pulp, and a portion is neutralised with alkali and concentrated. Further unneutralised waste liquor is then added, the mixture carbonised, and the residue extracted ; the extract is suitably treated to recover the sodium hydrogen sulphite.

A. Geake.

Treating waste sodium monosulphite liquors. V. Drewsen, Assr. to West Virginia Pulp and Paper Co. (U.S.P. 1,560,900, 10.11.25. Appl., 18.10.23. Renewed 4.4.25).—Waste sodium sulphite liquor from the digestion of vegetable fibrous material is concentrated to a water content of 50%, incorporated with nitre-cake and 10—30% of hydrated lime to minimise loss of organically combined sulphur, incinerated, and melted. The product contains sodium carbonate and calcium sulphide. After removing the sodium salts by leaching, the residue is treated to produce sulphur dioxide, which is converted into sodium sulphite for use in preparing further quantities of cooking liquor.

Production of organic acids from soda-cellulose waste liquor. P. Deriveau, E. de Fersen, F. Fiesohi, E. Lancesseur, A. Potel, and P. Watel (F.P. 563,747, 21.6.22).—The waste liquor, after removal of cellulose, is heated in closed vessels with oxygen or gases containing it, in the presence or absence of catalysts. The oxygen preferably contains a sufficient quantity of steam to prevent the liquor from drying. Suitable catalysts include platinum and similar metals ; chromium, copper, cobalt, manganese, aluminium, and nickel ; oxides of these metals or of iron ; aluminium, zinc, and phosphorus carbides ; metal chlorides and bromides ; and cerium, manganese, and lead salts. The catalysts may be used in their usual form or in colloidal solution, or may be deposited upon non-reacting material, such as kieselguhr, asbestos, pumice stone, and coke. The products include sodium formate, acetate, and oxalate.

L. A. Coles.

Sizing cellulose fibres. Paper-sizing process. J. A. De Cew, Assr. to Process Engineers, Inc. (U.S.P. 1,558,845–6, 27.10.25. Appl., [A] 7.6.24, [B] 14.7.25).—(A) Precipitation of the resin is delayed until the end of the beating operation. (B) Resin emulsions are added to the paper pulp and coagulated with colloidal aluminium hydroxide. When coagulation is complete, aluminium sulphate is added.

D. J. Norman.

Protecting wool, furs, and other materials against moths. W. Lommel and H. Münzel, Assrs. to Farbenfabr. vorm. F. Bayer und Co. (U.S.P. 1,562,510, 24.11.25. Appl., 10.5.24).—See G.P. 402,341 ; B., 1925, 202.

Preliminary treatment of cellulose prior to esterification. Soc. Chim. des Usines du Rhône (E.P. 237,567, 5.6.25. Conv., 28.7.24).—See U.S.P. 1,543,310 ; B., 1925, 667.

Utilising sulphite-cellulose [waste] lye. C. G. Schwalbe (U.S.P. 1,564,142, 1.12.25. Appl., 17.10.24).—See E.P. 224,509 ; B., 1925, 587.

Binding material for cement etc. (G.P. 399,020).—See IX.

Mixed glycerides. (U.S.P. 1,558,299).—See XII.

Acid and salts strongly absorbing ultra-violet rays. (E.P. 242,721).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of cellulose acetate silk. V. Kartaschov (Helv. Chim. Acta, 1925, 8, 928—942).—Cellulose acetate silk is not very permeable to water, it is far from porous, and exhibits no crystalline structure. It may be regarded as a solid colloid possessing a negative electrostatic charge. The electric charge does not, however, enter into the mechanism of dyeing. The silk may be dyed merely by immersion in a suspension of basic anthraquinone dyestuffs in water, suggesting that the mechanism is that of simple dissolution of dye by the colloidal fibre. This view has been tested by placing dry fibre and solid dry dyestuff in contact at 60° for several days. The fact that permanent dyeing ensues proves that the dyeing of cellulose acetate is a simple solution effect in which water may play a useful but not an indispensable part.

R. A. Morton.

Urine as a mordant in the dyeing of glove skins, and its replacement. E. Simoncini (Boll. Uff. Staz. Sperim. Ind. Pelli, 1925, 3, 269—276).—The putrefied urine commonly used as a mordant in the dyeing of glove leathers may be replaced with advantage by a solution containing about 0·3% of sodium ammonium phosphate and 2% of ammonium carbonate.

T. H. Pope.

Application of dyeing phenomena in the revelation of bleached written characters. R. Mellet and M. A. Bischoff (Compt. rend., 1925, 181, 868—869).—Ink texts bleached with oxidising agents may be revealed by staining the paper in a fluorescent dye solution (*e.g.*, eosin) and examining in filtered ultra-violet rays, *e.g.*, the light from a Wood lamp. The characters then appear plainly in a dark colour on a luminous coloured ground.

S. K. Tweedy.

Methods for obtaining white and multicolour effects on fabrics dyed with sulphur dyestuffs. A. Scheunert and J. Frossard (Sealed Notes [A] 1775, 18.10.07, [B] 1794, 16.12.07, [C] 2.1.08, and [D] 1813, 10.3.08. Bull. Soc. Ind. Mulhouse, 1925, 91, 551—557. Report by C. Zündel and X. Vogt, *ibid.*, 557—558). — (A) Resists under sulphur dyestuffs are obtained by means of printing pastes

containing manganese chloride and zinc oxide. White resist effects are produced by printing white mercerised cotton fabrics with a paste (*a*) containing 400 g. of manganese chloride, 500 g. of gum water, and 100 g. of zinc oxide, then drying, and dyeing in the usual manner for 5—30 secs. at a temperature not exceeding 70° with a dye liquor prepared with sulphur dyestuffs and sodium sulphide or sodium hyposulphite. Black and white effects are obtained by printing fabric with reserve paste *a* and a suitable Aniline-Black printing paste, the fabric being passed through a Mather–Platt before dyeing. Coloured effects obtained by means of ice colours are produced by printing the fabric, after preparation with β-naphthol, with a paste prepared by adding diazo-compounds or preferably their double salts with zinc chloride or aluminium sulphate to reserve paste *a*, drying, and developing the shade rapidly by passing the fabric during ¾ to 1 min. through a Mather–Platt, the fabric being subsequently dyed with sulphur dyestuffs. Satisfactory printing pastes for obtaining coloured effects by means of Para Red or Grenat N (Naphthylamine Claret) contain 100 g. of a double salt of diazotised *p*-nitroaniline and zinc chloride, 450 g. of reserve paste *a* and 450 g. of reserve paste *b*, or 150 g. of a double salt of diazotised α-naphthylamine and zinc chloride, 425 g. of reserve paste *a* and 425 of reserve paste *b* respectively. Reserve paste *b* contains 500 g. of zinc chloride and 500 g. of gum water. (B) Coloured resist effects are obtained under sulphur dyestuffs and also under vat dyestuffs such as Indanthrene Blue, Flavanthrene, Algol Blue, Algol Rose, Thioindigo Red, and Thioindigo Scarlet, by the method described in (A) by means of mordant (*e.g.*, Alizarine Blue and Alizarine Viridine) and basic (*e.g.*, Prune pure and Setocyanine) dyestuffs which are not immediately precipitated by zinc or/and manganese chloride or of which precipitation may be prevented by addition of suitable substances (*e.g.*, glacial acetic acid and phenol or resorcinol) but which are fixed on fabric by treatment for 1—2 min. in a Mather–Platt. Satisfactory printing pastes for blue and violet effects contain 30 g. of Setocyanine, 50 g. of glacial acetic acid, 100 g. of phenol, 700 g. of reserve paste *b*, 100 g. of a 50% solution of tannic acid in acetic acid of 6° B. (*d* 1·05), and 20 g. of Turkey-red oil, or 30 g. of Prune pure, 50 g. of glacial acetic acid, 100 g. of phenol, 700 g. of reserve paste *b*, 100 g. of chromium bisulphite, and 20 g. of Turkey-red oil respectively. (C) Especially bright red resist effects are obtained under Indanthrene Blue by means of the resist paste *a* described in (A), whereas inferior effects are obtained similarly under Indigo or blue sulphur dyestuffs. For example, cotton fabric is printed with the Para Red paste described in (A), then dried, dyed at 55—60° for 20—40 sec. in a dye-liquor containing Indanthrene Blue, and afterwards washed, acidified, washed, and soaped. These resist effects are quite satisfactory should several weeks elapse between printing and dyeing. (D) Coloured resist effects under sulphur dyestuffs are obtained simultaneously by means of ice colours and basic dyestuffs by the method described in (A), sodium tungstate being added to the β-naphthol used in the preparation of the fabric. Basic dyestuffs are maintained in solution in the reserve printing paste (containing zinc chloride) by addition of a mixture of lactic and glacial acetic acids or phenol. After printing, the fabric is dried, steamed twice in a Mather–Platt, dyed in a dye-bath containing sulphur dyestuffs and sodium sulphide, then washed, acidified, washed, and soaped. Zundel and Vogt report favourably on the processes, although they find that the presence of zinc chloride in the reserve paste described in (C) retards fixation of basic dyestuffs.

A. J. HALL.

Fastness of dyestuffs on wool. HIRST.—See IV.

PATENTS.

Dyeing, printing, or stencilling acetylcellulose. BRITISH CELANESE, LTD., G. H. ELLIS, and W. O. GOLDTHORPE (E.P. 242,711, 14.8.24; cf. E.P. 219,349, 224,681, 227,183 and 237,943; B., 1924, 906; 1925, 39, 204, 801).—The dyeing of cellulose acetate with insoluble dyes and fatty substances, such as sodium sulphoricinoleate, is aided by the presence of secondary solvents; these may be alkyl halides, such as tetrachloroethane and trichloroethylene, aromatic compounds, such as cresols, alkylanilines, toluidines, chlorophenols, and chlorobenzenes, and hydrogenated aromatic substances, such as hexahydrophenol, hexahydrocresols, hexahydrobenzene, decahydronaphthalene, and tetrahydronaphthalene. The presence of such solvents results in improved depth of shade, penetration, and levelness, and consequently in economy in dyes. The mixture of dye, oil, and solvent may be made in any order, much or little water may be used, and the mixture may be subsequently concentrated or dried before addition to the dye bath or printing paste. Insoluble diazotisable substances may be applied in this way for development on the fibre, and the process may be used for the production of varied effects on mixed goods. (Reference is directed, in pursuance of Sect. 8 (2), of the Patents and Designs Acts, 1907 and 1919, to E.P. 224,925.)

A. GEAKE.

Treating textile fibres. J. C. MCDOWELL (U.S.P. 1,558,104, 20.10.25. Appl., 8.5.22).—Cotton or linen goods dyed in part with vat colours are boiled under pressure at 120° with a solution of sodium silicate and sodium carbonate. The undyed portions of the fabric can then be bleached by one of the usual methods without the coloured part being affected. T. S. WHEELER.

Kiers. P. F. HADDOCK (E.P. 243,262, 15.6.25).

Azo-dyestuffs (U.S.P. 1,558,890). See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Catalytic oxidation of ammonia under works conditions. W. J. MALIAREVSKI and N. A. MALIAREVSKAJA (Z. angew. Chem., 1925, 38,

1113—1118).—The catalytic oxidation of ammonia gas with a set of platinum gauzes was investigated for the determination of the relation between: (1) ammonia concentration and temperature, and (2) nitric oxide yield and temperature, each with varying gas velocities. The results which are tabulated in detail and also exhibited as curves, are in agreement with those of other investigators. The optimum temperature for nitric oxide yield, as measured in the gas leaving the gauze with a Le Chatelier pyrometer, was found to be 620°. This optimum rises and falls with the gas velocity. At temperatures above the optimum the yield falls off rapidly with low gas velocities. The ammonia concentration representing optimum conditions in the authors' apparatus was 9·2% by vol. and the time of contact 0·00031—0·00044 sec. C. Irwin.

Action of silica on barium and magnesium sulphates. G. Marchal (Compt. rend., 1925, **181**, 784—786; cf. B., 1924, 92).—When heated alone barium sulphate begins to decompose at 1510°, but when mixed with the theoretical amount of silica, the decomposition begins at 1025° according to the reaction, $BaSO_4+SiO_2\rightleftharpoons BaO,SiO_2+SO_2+\frac{1}{2}O_2$. For magnesium sulphate the corresponding temperatures are 880° and 680° respectively. The equilibrium pressures have been determined from 1100° to 1270° for barium sulphate, and from 700° to 1030° for magnesium sulphate. The heats of formation of barium and magnesium silicates, from the oxide and silica, are calculated as 14·7 and 8·5 cal. respectively. W. Hume-Rothery.

Apparatus for the determination of helium in natural gas mixtures and minerals. W. Chlopin and A. Lukasuk (Ber., 1925, **58**, 2392—2396).—The method depends on the absorption of gases other than helium by coconut charcoal. The apparatus consists of two parts separated by a glass tap. One portion comprises the absorption vessel filled with coconut charcoal, a manometer, and a small Plücker tube for spectroscopic examination of the unabsorbed gas, and the other is a somewhat modified form of the McLeod vacuometer and contains the measuring tube. The process requires only 100—200 c.c. of gas. Test analyses give results in agreement with the data obtained by the method of Moureu and Lepape (J., 1914, 1118). H. Wren.

Foreign elements in scheelite and tungstic acid. Agte, Becker-Rose, and Heyne.—See X.

Precipitation of calcium sulphite. Farnell.—See XVII.

Patents.

Manufacture of hydrocyanic acid. Deutsche Gold- u. Silber-Scheideanstalt vorm. Roessler, and O. Liebknecht (E.P. 242,685, 14.6.24. Addn. to 207,830; B., 1925, 447).—The efficiency of an activated carbon catalyst in promoting the interaction of ammonia and carbon monoxide to yield hydrogen cyanide is increased by the presence of alkali. A mixture of coal powder and sawdust may be treated with concentrated caustic soda solution, dried, and ignited at 800° in contact with the mixture of reacting gases. After carbonisation the temperature is lowered to 550—600° and the reaction proceeds. Other alkalis may be used. (Reference is directed in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 207,830.) C. Irwin.

Oxidation of ammonia. C. Ellis, Assr. to Ellis Foster Co. (U.S.P. 1,558,598, 27.10.25. Appl., 26.4.18. Renewed 13.1.25).—Air with 5% of ammonia is passed over a catalyst composed of cobalt vanadate mounted on a carrier maintained at a temperature of about 600°. A good yield of nitrogen oxides is obtained. The catalyst is prepared by mixing cobalt chloride with ammonium vanadate and heating to volatilise ammonium chloride. The vanadates of nickel, chromium, and manganese may also be used. T. S. Wheeler.

Apparatus for synthetic production of ammonia from its elements. A. Woosnam. From Norsk Hydro-Elektrisk Kvaelstofaktieselskab (E.P. 222,461, 2.9.24).—A catalyst chamber suitable for use with very active catalysts at somewhat low temperatures consists of an outer pressure-resisting shell and an inner chamber containing the catalyst and having walls pervious to gas. This may be built up of rings bolted together or consist of two perforated tubes with the space between them packed with loose material. Both inner and outer chambers are fitted with gas connexions, and the temperature of the catalyst is controlled by regulating the proportion of the gas mixture which passes through the porous wall of the inner chamber into the outer space. A heating element may be arranged in this outer space or in a central tube in the inner chamber, or the apparatus may be started by admitting the gas into the inner chamber and burning it with air in the outer. C. Irwin.

Treatment of natural alkaline salts. G. M. Clark. From Cosmic Arts, Inc. (E.P. 217,598, 12.6.24).—Trona or other natural salts containing a substantial proportion of sodium carbonate are dissolved in water, and the solution is concentrated, or similar natural alkaline waters are concentrated, and then treated with ammonia and carbon dioxide at 33°. The whole of the sodium carbonate present, about two-thirds of the sodium chloride, and some of the sodium sulphate are converted into sodium hydrogen carbonate, which is precipitated and is filtered off and washed at the same temperature. C. Irwin.

Production of practically iron-free alumina. Chem. Fabr. Griesheim-Elektron (E.P. 241,184, 30.9.25. Conv., 13.10.24. Not yet accepted).—Clay or the like is treated with hydrochloric acid or with sulphuric acid followed by sodium chloride, so as to obtain a crude solution of aluminium chloride. The iron is reduced, for instance, by hydrogen sulphide, and the solution is then evaporated and the residue calcined to decompose the aluminium chloride while excluding oxygen so that the ferrous

chloride is not decomposed. The solution may be sprayed into a tower or rotary tube furnace where it comes in contact with hot gases having a reducing character, obtained for instance by flameless combustion. The residue is leached with water containing hydrochloric acid, which dissolves the ferrous chloride, leaving alumina practically free from iron. The alumina may be further purified by heating in a current of air containing chlorine or hydrochloric acid to remove the remaining iron.

Process for making hydrated alumina and by-products. G. L. WILLIAMS (U.S.P. 1,559,489, 27.10.25. Appl., 29.12.23).—Potassium aluminate solution is treated with boiler furnace gases, whereby hydrated alumina is precipitated in a granular form.
H. ROYAL-DAWSON.

Synthetic manufacture of liquid ammonia. G. PATART (E.P. 240,871, 5.10.25. Conv., 6.10.24. Not yet accepted).—The cold produced by the evaporation of the liquid product of the catalytic synthesis of ammonia is used for cooling the gases withdrawn by the compressor used for producing the pressure necessary for the ammonia synthesis, thereby increasing the output of the compressor; or for cooling the gaseous components or a mixture of the components for purification purposes; or for effecting a preliminary cooling of the nitrogen and hydrogen before their separation by liquefaction and rectification from complex gas mixtures containing them. The liquid ammonia may further be utilised as washing liquid for the final purification of the compressed gases, whereby carbon moxoxide, water vapour, hydrogen sulphide, etc. are removed. When the cold produced by the evaporation of the liquid ammonia is used for purifying the gases, an absorbent is chosen the efficiency of which increases with a reduction of temperature; *e.g.*, gaseous hydrocarbons are absorbed by coal oils or activated carbon, carbon dioxide by water under pressure or alkali carbonates, and carbon monoxide by cuprammoniacal and like solutions.

Treatment of leucite and other similar complex silicates containing potassium. W. R. ORMANDY and A. M. PEAKE (E.P. 242,336, 2.8.24 and 29.4.25).—Leucite is allowed to react with one or more alkaline-earth salts of phosphoric acid or a natural phosphate rock, calcium carbonate, and dilute sulphuric acid or an acid sulphate, in the presence of water, the ingredients being finely ground and intimately mixed. The reaction is exothermic, and the degree of heat developed must be controlled. Practically the whole of the potassium present can be recovered in the form of neutral salts substantially free from iron and aluminium, and the insoluble phosphate is converted into a form in which it is soluble either in water or in weak citric acid or ammonium citrate solutions. By varying the quantities of the reacting substances, and controlling the temperature of the reaction, the composition of the product as regards solubility of the potash and phosphate values in water or in weak citric acid solution, can be varied at will. The quantity of sulphuric acid required for the reaction is not more than twice the amount theoretically required for the potash present. Before, during, or after the reaction, substances having value as fertilisers may be added.
M. E. NOTTAGE.

Stabilising bleaching powder. A. LAMBLE, and UNITED ALKALI CO., LTD. (E.P. 242,805, 15.12.24). —Bleaching powder is dried, for instance by controlled heating on trays exposed to the atmosphere, or by the action of dry air free from carbon dioxide, and a small quantity of powdered quicklime added. The bleaching powder is thus stabilised without appreciable reduction of its available chlorine content.
C. IRWIN.

Combination apparatus for the slaking and mixing of lime, particularly for use in sulphate of ammonia plant. R. AND J. DEMPSTER, LTD., and A. L. HOLTON (E.P. 242,865, 26.3.25).—An open basket with perforated bottom is mounted on, and revolves with, a central vertical shaft. On this basket lump lime is slaked and the cream falls into a tank wherein it is agitated by paddles carried by the downward extension of the shaft. In the case of lime intended for ammonia distillation the slaking will be by means of spent liquor, and the contents of the tank are further agitated by steam.
C. IRWIN.

Production of zinc sulphate from iron ores containing zinc sulphide. GEWERKSCHAFT SACHTLEBEN, and H. PÜTZER (G.P. 416,104, 5.2.24). —The residual roasted ore containing zinc sulphide is treated with vaporised sulphuric acid or a mixture of sulphur trioxide and steam at a temperature above that of the formation of iron sulphate and below the decomposition temperature of zinc sulphate· preferably at about 600°; if desired the counteri current principle may be used in the process. Alkali sulphates, bisulphates, or pyrosulphates, or other alkali salts which when heated with sulphur trioxide or sulphuric acid decompose with the formation of sulphates, bisulphates, or pyrosulphates, may be added to the roasted ore. The formation of zinc, silicate or zinc ferrite, which diminishes the yield of zinc sulphate, is prevented.
J. S. G. THOMAS.

Treatment of alkaline-earth sulphides. RHENANIA VEREIN CHEM. FABR. A.-G. MARWEDEL, and W. SCHOLZ (G.P. 417,410, 2.2.24).—Alkaline-earth (*e.g.*, barium) sulphide solutions are treated with ferric or ferrous hydroxide, or with material containing them, in such a manner that double alkaline-earth–iron compounds soluble in water are formed, and these are subsequently treated with hydrochloric or other acid in such quantities that solutions free from iron are obtained, or, alternatively, an excess of the acid is used, and the solution is subsequently freed from iron by the addition of barium silicate.
L. A. COLES.

Production of barium salts. RHENANIA VEREIN CHEM. FABR. A.-G. ZWEIGNIEDERLASSUNG MANNHEIM, Assees. of F. RÜSBERG and A. KLÜG (G.P. 417,441, 28.9.23).—Suspensions of solid barium sulphide in salt solutions, *e.g.*, natural brine,

are treated with hydrogen sulphide, preferably with heating. L. A. Coles.

Simultaneous production of barium chloride and sulphur chloride. Chem. Fabr. Griesheim-Elektron, Assees. of K. L. Gonder (G.P. 417,853, 17.12.22).—The compounds are produced by passing chlorine over strongly heated mixtures of barium sulphate with coal, coke, or hydrocarbons with a high carbon content. L. A. Coles.

Production of hydrides. E. Tiede (G.P. 417,508, 23.1.25).—The compounds are prepared by heating alkali, alkaline-earth, or rare-earth azides in a stream of activated hydrogen. L. A. Coles.

Production of sodium thiosulphate. P. Kircheisen (G.P. 417,602, 31.7.24).—Sodium hydrosulphide solution, produced from barium sulphide solution by treatment with sodium bisulphate or sodium bicarbonate, or with equimolecular proportions of the free acids and the normal salts, is oxidised to sodium thiosulphate. L. A. Coles.

Production of barium salts. J. Ephraim (G.P. 418,097, 23.4.22).—Hydrogen sulphide is passed into a heated suspension of solid barium sulphide in a concentrated solution of barium sulphide, and the resulting solution caused to react with solid alkali salts. J. S. G. Thomas.

Stabilising solutions containing loosely-combined oxygen [hydrogen peroxide]. Stabiliser for solutions containing loosely-combined oxygen. V. Wintsoh, jun., Assr. to R. H. Comey Co. (U.S.P. 1,559,599–600, 3.11.25. Appl., 25.6.24).—(A) 1% of sodium pyrophosphate salicylate (see B) acts as a stabiliser when added to solutions containing loosely-combined oxygen, *e.g.*, a solution of hydrogen peroxide. (B) Sodium pyrophosphate is mixed with salicylic acid moistened with water, and the mixture dried on the water bath. Sodium pyrophosphate salicylate, for which the formula $(NaO{\cdot}CO{\cdot}C_6H_4)_4P_2O_7$, is suggested, is thus obtained. T. S. Wheeler.

Method of producing an allotropic form of sulphur. H. H. Wilkinson, Assr. to Union Sulphur Co. (U.S.P. 1,560,926, 10.11.25. Appl., 20.12.23).—Sulphur is heated under vacuum in a still until it melts and vaporises. The vapour, still under vacuum, is drawn into a vacuum receiver with the aid of a current of a non-oxidising gas inert to sulphur, and there condensed. The finished product consists of finely divided, deep yellow, highly purified, light-weight sulphur composed of particles of approximately equal size, and free from sulphur dioxide. M. E. Nottage.

Preparing adsorptive silicious material. F. X. Govers (E.P. 243,123, 25.9.24).—See U.S.P. 1,504,549 and 1,506,118; B., 1924, 829, 870. Iron, silver, platinum, or other catalysts in colloidal form may be added to the colloidal silicic acid.

Purification of phosphoric acid containing arsenic. O. Wolfes and H. Maeder (U.S.P. 1,562,818, 24.11.25. Appl., 5.5.25).—See E.P. 234,122; B., 1925, 630.

Separating potassium and sodium hydroxides. T. Sutter, Assr. to Soc. of Chem. Ind. in Basle (U.S.P. 1,562,805, 24.11.25. Appl., 8.5.25).—See E.P. 234,072; B., 1925, 630.

Production of potassium carbonate. H. Klopstock and W. Neumann (U.S.P. 1,562,891, 24.11.25. Appl., 21.7.24).—See E.P. 234,585; B., 1925, 590.

Combustion reactions. (E.P. 242,681).—See I.

Colloidal suspensions. (E.P. 242,689).—See I.

Gas analysing method. (U.S.P. 1,560,660).—See II.

Cyanide solutions. (U.S.P. 1,562,295).—See X.

VIII.—GLASS; CERAMICS.

Colouring glass with phosphates. K. Fuwa (J. Jap. Ceram. Assoc., 1924, 32, 431—440).—In the glasses $R_2O,RO,3SiO_2$ variation in the kind of alkali has no effect on the opalescence produced by bone ash. Lead-glasses give the best effect, followed by barium-glasses and then by zinc-glasses. Boric acid favours the action of bone ash. RO has a remarkable influence on the coloration of the borosilicate glasses $R_2O,RO,0{\cdot}5B_2O_3,3SiO_2$; ZnO favours the action of bone ash most, followed by BaO and PbO, MgO-glasses showing the least effect. Experiments with the glasses $1{\cdot}3$—$1{\cdot}7R_2O,RO,6SiO_2$ and $R_2O,B_2O_3,6SiO_2$ showed that the borosilicates require the least amount of bone ash for opalescence, followed by lead. and barium-glasses; calcium glasses are least effective in this respect. Disodium phosphate acts similarly to bone ash, but in the glasses $R_2O,RO,0{\cdot}5B_2O_3,3SiO_2$ the boric anhydride has an unfavourable effect. With tricalcium phosphate, in glasses of the type $R_2O,RO,3SiO_2$, PbO favours the opalescence most, followed by BaO; B_2O_3 favours it. In the glasses $R_2O,RO,0{\cdot}5B_2O_3,3SiO_2$, PbO and BaO favour the action of the phosphate, ZnO being less active in this respect. The action of disodium phosphate is weaker than that of bone ash and tricalcium phosphate in the glasses $R_2O,RO,0{\cdot}5B_2O_3,3SiO_2$, whereas no notable difference is observable in the glasses $R_2O,RO,3SiO_2$. The opalescence is probably due to suspended phosphorus trioxide. Potassium nitrate and arsenious oxide seem to favour slightly the action of the phosphates. S. Kondo.

Colouring glass with arsenious acid. K. Fuwa (J. Jap. Ceram. Assoc., 1924, 32, 469—470).—Experiments with glasses of the types $1{\cdot}3R_2O,RO,6SiO_2$ and $R_2O,B_2O_3,6SiO_2$, using amounts of arsenious acid varying from 5 to 25%, showed that a large quantity of arsenious acid, *e.g.*, 15%, produces an opalescence on melting the batches or on reheating the glasses. The quantity of the acid required to produce this effect varies according to the composition of the glass. The effect is due probably to the presence of suspended arsenious oxide. Glasses

containing arsenious acid are coloured brown on the surface when heated in a flame, the effect being more marked in potash-glasses than in soda-glasses.
S. KONDO.

Colour imparted to glass by carbon and its compounds. K. FUWA (J. Jap. Ceram. Assoc., 1924, 32, 471—490).—Experiments with glasses of the types $R_2O,RO,3SiO_2$, $R_2O,RO,0{\cdot}5B_2O_3,3SiO_2$, $1{\cdot}3$—$1{\cdot}7R_2O,RO,6SiO_2$, and $R_2O,B_2O_3,6SiO_2$ showed that retort carbon and organic compounds (sucrose, tartaric and oxalic acids) produce a colour which varies from light yellow or brown to dark red. Carbon in excess floats on the surface of the glass or remains suspended in it, imparting a dark colour. A dark colour is produced in calcium-, magnesium-, and barium-glasses, whereas zinc- or lead-glasses remain colourless or acquire only a lighter colour. Soda glasses give a lighter colour than potash-glasses, but the content of alkali has no material influence provided that the constituents of the glasses remain unchanged. The colouring power of organic compounds increases with their content of carbon and decreases with their content of oxygen and hydroxyl. In soda-glasses, the coloration is destroyed by addition of potassium nitrate in quantities about 10—20 times as much as the carbon, but potassium nitrate only weakens the coloration in potash-glasses. The coloration in some soda-glasses is destroyed by arsenious acid in quantity about 3–5 times as much as the carbon, but most potash-glasses retain the colour though it may be weakened to some extent. S. KONDO.

Ray-transmission of glass. I. Transparency to ultra-violet rays of alkali-lime glasses, and manufacture of glasses which transmit ultra-violet rays. J. SUGIE (Osaka Ind. Lab. Bull., 1924, 5, [11], 26 pp.).—Tests were made with 51 glasses of the type $0{\cdot}7$–$1{\cdot}6Na_2O$, $0{\cdot}9$–$1{\cdot}5CaO$, $6SiO_2$, 9 glasses obtained by replacing the whole or a part of soda by potash, and 17 glasses made by adding MgO, Al_2O_3, B_2O_3, As_2O_3, MnO_2, P_2O_5, Sb_2O_3, WO_3, Fe_2O_3, ZnO, BaO, SnO, CdO, TiO_2, or ZrO_2. The glass $1{\cdot}0Na_2O,1{\cdot}4CaO,6SiO_2$ was most transparent to ultra-violet rays, the minimum wave length of the transmitted rays being 237 $\mu\mu$ for a plate 1·50 mm. thick. The glass $1{\cdot}0Na_2O,1{\cdot}5CaO,6SiO_2$ was the least transparent, the corresponding minimum wave-length being 279$\mu\mu$. The increase of the transparency with diminution of thickness is smaller in the less transparent glasses than in the more transparent kinds. The effect on the transparency of replacing soda entirely or partially by potash is negligible. Phosphorus pentoxide does not injure the transparency of the alkali–lime glasses, TiO_2, Sb_2O_3, and Fe_2O_3 have a marked effect on the transparency, and MgO and Al_2O_3 have also some effect. S. KONDO.

[Ceramic bodies for] ignition-plugs. M. WATAYA (Osaka Ind. Lab. Bull., 1924, 5, [8] ; 30 pp.).—Analysis of an ignition plug of German manufacture (Bosch) gave loss on ignition 0·11, SiO_2 66·43, Fe_2O_3 2·01, Al_2O_3 3·72, CaO trace, MgO 27·78, K_2O 0·02, Na_2O 0·02%, and TiO_2 trace. It was coated with a slip containing rutile and with a colourless glaze. On the basis of this analysis 27 bodies were prepared. Thick tubes were pressed semi-dry, dried and biscuit-fired at 900–1000°. They were coated with slips, prepared from the same bodies to which 1·5% of rutile had been added, and were glost-fired at 1350—1520°. A glaze, ($0{\cdot}33Na_2O$, $0{\cdot}33CaO$, $0{\cdot}33PbO$), $0{\cdot}13Al_2O_3$, ($1{\cdot}73SiO_2$, $0{\cdot}53B_2O_3$), maturing at 1100°, was then applied on the tubes. The finished plugs were tested for resistance to sudden cooling and heating, electrical resistance at high temperatures, and puncture voltage, and the microstructure was examined. The results indicate that limestone is not a suitable flux for the body. Clay acts as a strong flux in magnesian bodies. Porcelains made from bodies containing Shugan-stone (loss on ignition 5·16, SiO_2 44·60, Al_2O_3 0·57, Fe_2O_3 0·91, CaO 0·25, MgO 47·82) have strong electrical resistance at high temperatures, but do not resist sudden changes in temperature. Bodies made of 80 pts. of calcined talc and 10—15 pts. of clay substance are best for talc plugs. Kaolin is better than a plastic clay with respect to resistance to sudden changes in temperature. S. KONDO.

Iron aventurine glaze. S. KONDO (Tokyo Higher Tech. School, Bull. 3, 1925, 1—12).—In fritted glazes of the composition ($0{\cdot}5Na_2O$, $0{\cdot}25BaO$, $0{\cdot}25$ CaO)($0{\cdot}05$ Al_2O_3, $0{\cdot}5$—$0{\cdot}1$ Fe_2O_3) ($2{\cdot}00$—$2{\cdot}80$ SiO_2, $1{\cdot}00$—$0{\cdot}20$ B_2O_3), silica retards the production of the aventurine, and boric oxide favours it, though excess makes the crystals coarse ; the best proportions for cone 05a—1a are $2{\cdot}00$ SiO_2 : $0{\cdot}20$ B_2O_3. At least $0{\cdot}30Fe_2O_3$ is required. In fritted glazes of the composition ($0{\cdot}5$ Na_2O, $0{\cdot}25BaO$, $0{\cdot}25$ CaO)($0{\cdot}05$—$0{\cdot}25$ Al_2O_3, $0{\cdot}50$—$0{\cdot}10$ Fe_2O_3), ($2{\cdot}50$ SiO_2, $0{\cdot}50$ B_2O_3), alumina retards the crystallisation, but keeps the crystals fine and widens the permissible range of ferric oxide. In fritted glazes of the composition ($0{\cdot}50$ Na_2O, $0{\cdot}25$ BaO, $0{\cdot}25$ CaO) ($0{\cdot}05$ Al_2O_3, $0{\cdot}50$ Fe_2O_3) ($2{\cdot}00$ SiO_2, $0{\cdot}50$ B_2O_3) metallic iron is the most effective source of iron, but the crystals are not well defined and are contaminated with amorphous material. Ferrous carbonate is next in order of effectiveness and produces good crystals. Ferrous oxide is less effective, and ferric oxide least. In a series in which R_2O, RO, and Fe_2O_3 were varied, the best glazes were obtained with ($0{\cdot}25$ K_2O, $0{\cdot}25$ Na_2O, $0{\cdot}50$ CaO) ($0{\cdot}05$ Al_2O_3, $0{\cdot}45$—$0{\cdot}50$ Fe_2O_3)($2{\cdot}00$ SiO_2, $0{\cdot}20$ B_2O_3), fired to cone 03a and cooled quickly. Ferruginous bodies favour the crystallisation and the glazes appear finer than when applied on porcelain bodies. Excess of the colouring oxides (CaO, CuO, and UO_3) makes the crystals too minute and injures the lustre. Fine glazes were obtained with UO_3. In a series with varying Al_2O_3 the best result was obtained with ($0{\cdot}25$ K_2O, $0{\cdot}25$ Na_2O, $0{\cdot}50$ CaO) ($0{\cdot}05$ Al_2O_3, $0{\cdot}45$ Fe_2O_3) ($2{\cdot}00$ SiO_2, $0{\cdot}20$ B_2O_3).
S. KONDO.

Mechanical analysis of sediments. COUTTS and CROWTHER.—See I.

Size distribution of particles. WERNER.—See I.

Electrical conductivity of magnesia refractories at high temperatures. Burt-Gerrans and Kerr.—See XI.

Patents.

Glasses. Jenaer Glaswerk Schott u. Gen., O. Schott, and H. Thiene (E.P. 242,568, 4.8.25).—A glass having a relatively high softening temperature and insensitive to abrupt temperature changes contains at least 45% of silica, 2—15% of boron trioxide, and 4—30% of lime and magnesia together. The alkali oxides usually present as a flux are largely replaced by alumina, which is present to the extent of 20—30%, against 4—8% of alkali oxides. Lead oxide and antimony oxide together may be added up to 6%. B. W. Clarke.

[Glass] melting furnace. W. A. Yung (U.S.P. 1,561,393, 10.11.25. Appl., 15.4.25).—A rectangular furnace for glass melting is divided into two chambers by a longitudinal wall reaching from the bottom to the roof. Openings through the wall adjacent to one end of the furnace allow glass and the products of combustion to pass between the chambers. The lower portion of the dividing wall is thickened to form relatively narrower spaces for the flow of the molten glass than for the products of combustion. The outer walls of the furnace are offset outwards between their upper and lower edges and the dividing wall is provided with a cooling system.
B. W. Clarke.

Silvering glass or other surfaces of non-conconducting material. Deutsche Gasglühlicht-Auer-Ges.m.b.H. (G.P. 417,787, 19.3.22).—The surface to be silvered is first cleaned with a dilute solution of formaldehyde and subsequently treated with the usual silvering solution.—A. R. Powell.

Purifying [quartz] fusions. E. Thomson, Assr. to Gen. Electric Co. (U.S.P. 1,559,203, 27.10.25. Appl., 1.6.25).—Fused quartz is centrifuged in a heated crucible so that it forms a hollow cylinder to the inner surface of which the air bubbles present gravitate. When the rotation is stopped, the portion containing the air bubbles rises to the surface of the fused mass and is removed when cold. Clear quartz is thus obtained.
T. S. Wheeler.

Treating ceramic mixtures. H. Spurrier (U.S.P. 1,559,652, 3.11.25. Appl., 24.1.24).—The porosity of plastic clay is reduced by placing the clay in a container which is evacuated and to which air is then suddenly admitted. T. S. Wheeler.

Purifying clays and improving their colour. W. Feldenheimer (E.P. 242,357–8, 7.8.24).—(a) Clay suspended in water is treated with a water-soluble reducing acid, *e.g.*, oxalic acid, and a soluble salt of a reducing acid, such as sodium sulphide or sodium thiosulphate, a permanent bleaching effect being produced. The clay may be purified by deflocculation if the sodium sulphide solution is added to the clay slip before the oxalic acid. (b) Clay suspended in water is brought into contact with an acid salt of sulphurous acid, such as sodium hydrogen sulphite or sodium metabisulphite, and a metal, such as zinc, which reduces sulphurous acid and does not form coloured oxidised salts. The reaction is accelerated by adding a trace of a mineral acid, *e.g.*, hydrochloric acid, which gives the less readily oxidised iron salts.
B. W. Clarke.

Recovering diatomaceous earth from waste material [from sugar refining]. R. C. Williams (U.S.P. 1,561,042, 10.11.25. Appl., 15.7.25).—Waste decolorising earth from sugar refineries is mixed with an antiseptic such as phenol, to prevent fermentation and is then used to make moulded articles. Alternatively the earth is mixed with water and allowed to ferment. When fermentation is complete any gas in the material is removed and the earth used as above. T. S. Wheeler.

Preparation of porous bodies of magnesia. Deutsche Ton- u. Steinzeug-Werke A.-G. (G.P. 416,901, 2.7.21).—Magnesium silicate is mixed with magnesium carbonate or hydroxide or with other magnesium compounds which yield the oxide on heating. The products are plastic and when fired are porous and resistant. The mixture is suitable for making diaphragms. A. R. Powell.

IX.—BUILDING MATERIALS.

Patents.

Paste for admixture with cement and other materials for strengthening and/or waterproofing them. S. McM. Kirkpatrick (E.P. 242,345, 6.8.24).—A paste consisting of crude or vulcanised rubber latex, mixed with hexamethylenetetramine or other preservative agents, sodium silicate, potash soap, and water, is added in the proportion of 3½ lb. per 100 lb. of cement, and acts as a strengthening and waterproofing agent. Bricks, blocks, tiles, etc. may be waterproofed by dipping in a fluid suspension of the paste in water.
B. W. Clarke.

Production of finely-crushed diabases, granites and allied stones. E. B. Hack and E. J. Burt (E.P. 242,487, 6.2.25).—The crushed material is passed successively through two tubular containers with their axes inclined to the horizontal and to one another, and provided with means for raising and dropping the material during its passage. The material is dried in a current of hot air or gas in the upper container, and is delivered to the lower container, where it meets a stream of cool air which separates out the finely-crushed material. The product is for use in road-making and in cement and concrete mixtures. B. W. Clarke.

Preparation of a binding material for cement, mortar, and the like. H. Silbermann (G.P. 399,020, 8.1.21).—A strong binding material for cement and the like is made by treating waste cellulose liquors, either alone or mixed with sodium silicate, alumina, sodium carbonate, or resin soap,

with oxidising agents, such as bleaching powder or peroxides. The products are resistant to weathering. A. R. Powell.

Preserving wood. J. H. James, Assr. to C. P. Byrnes (U.S.P. 1,561,164, 10.11.25. Appl., 19.12.21).—Wood is impregnated with a liquid preservative containing alcohols, aldehydes, and aldehyde-fatty acids obtained by the partial oxidation of petroleum hydrocarbons (cf. E.P. 138,113 and 209,128; J., 1921, 636 A; B., 1924, 210). B. W. Clarke.

Composition of matter. [Artificial stone.] H. S. Lukens, Assr. to Solidifier Corp. (U.S.P. 1,561,473, 17.11.25. Appl., 24.1.25).—An aggregate, other than metallic ores, is mixed with a binder consisting substantially of magnesia and water, and the wet mixture is carbonated to convert the magnesia. B. W. Clarke.

Making cement. A. G. Croll, Assr. to Atlas Portland Cement Co. (U.S.P. 1,562,207, 17.11.25. Appl., 3.3.24).—The raw materials are burnt at a clinkering temperature, and the clinker is quenched at a white heat. The Portland cement produced on grinding has a higher strength than the normal product. B. W. Clarke.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Use of a blast enriched in oxygen in the Thomas process. J. Haag (Stahl u. Eisen, 1925, **45**, 1873—1878).—Large-scale experiments have been carried out on the plant of the Thomaswerk der Gutehoffnungshütte in Oberhausen on the practicability of adding oxygen to the blast, both from the metallurgical and economic standpoints. A regulated measured supply of oxygen was sent into the furnace at about 4 atm. pressure. The waste gases were sampled at half-minute intervals and analysed. Temperatures of the pig iron, the steel, and the converter linings were measured by the Holborn-Kurlbaum pyrometer. The charges (kept as uniform as possible), the limestone used, and the slags were accurately weighed. During each operation a similar set of conditions (with regard to temperature etc.) was maintained. The use of oxygen in the blast was characterised by a dazzling white flame. Changes in this showed the transition to the de-phosphorisation stage. The product showed no deterioration in quality and could be poured and worked up by the usual methods. In order to avoid increased ferrous oxide in the crude steel, only air was used in the later stages of blowing. With 21·0% of oxygen in the blast the blowing period is 13·25 mins.; with 50% this becomes 5·56 mins. The product of the time of blowing in mins. and the percentage of oxygen in the blast is a constant. The higher temperature of the molten charge due to the diminution of the nitrogen ballast allows larger amounts of scrap to be used. Thus for a charge of 23,900 kg. with 21% of oxygen in the blast the amount of scrap was 600 kg., with 26% 2200 kg., with 35·1% of oxygen 3700 kg. The oxidation losses do not differ much from those with normal blast. Figures are given, showing the economies attained owing to the use of larger amounts of scrap and to the shortening of the blowing period. A shortening of 5·55 min. results when the oxygen content is raised from 21% to 35·1%. The slag is poorer in phosphorus than ordinarily, owing to the decrease in the amount of pig iron in the charge. For a charge of 23,900 kg. and a blast containing 35·1% of oxygen, the volume of oxygen required is 625 cub. m., the cost of which should not be higher than 4·36 pfennige per cub. m. From the thermal point of view the same results are obtained by using pig iron with 0·5% of phosphorus and adding 600 kg. of scrap iron, or with 1·85% of phosphorus and adding 3500 kg. of scrap iron. The use of an oxygen-enriched blast allows pig iron of lower quality, or poor in phosphorus to be used, and the ores used for the pig iron itself may show wide variation in their phosphorus content. A. Coulthard.

Production of press-matrix iron in the converter. A. Jung (Stahl u. Eisen, 1925, **45**, 1915—1917).—Press-matrix iron should have a tensile strength of 44—52 kg./mm.^{2} and should be neither too hard nor too tough. It is subjected to hammering and expanding tests to ensure freedom from brittleness. So as to be capable of being threaded it has a relatively high phosphorus content, which varies, however, with the thickness of the casting. The crystalline structure is not important so long as the properties are correct. The material is produced by stopping the blow during the dephosphorisation period. The manganese content must be sufficient to prevent red-shortness. The iron should not be overheated and should be poured at a relatively low temperature. The permissible range of phosphorus is 0·28—0·50% and the manganese content 0·30—0·70%. If the manganese content of the mixer iron is less than 1·50% there is a danger of red-shortness. The lime additions are less than when making ingot iron. Press-matrix iron should be blown without ferromanganese additions. The time from taking the test sample to emptying the charge should be as short as possible, but the tendency to re-phosphorisation is small. T. H. Burnham.

Corrosion of cast irons in sulphuric acid of varying concentration. G. Delbart (Compt. rend., 1925, **181**, 786—788; cf. B., 1925, 592).—The loss in weight of different kinds of cast iron in sulphuric acid has been determined, the concentration varying from 1·6 to 92·6% total SO_3. Phosphoric or impure cast irons are more rapidly attacked than pure or malleable cast irons, the difference being greatest for dilute acids. Cast irons are more rapidly attacked than cold-drawn steel in dilute acid, but in concentrated oleum the results are comparable, and some cast irons may even be the better. W. Hume-Rothery.

Chemical and physical processes in rusting and corrosion. O. Bauer (Gas- u. Wasserf., 1925, **68**, 683—687, 704—707).—A review of the

subject, with special reference to the work of the author and colleagues (cf. Heyn and Bauer, J., 1908, 569; 1910, 568; Bauer and Wetzel, 1916, 1112; Bauer and Vogel, 1919, 1444A; Bauer, B., 1925, 674). A. COULTHARD.

Origin of pitting corrosion phenomena [in iron water pipes]. E. LIEBREICH (Korrosion u. Metallschutz, 1925, 1, 67—69; Chem. Zentr., 1925, 96, II., 2024).—The formation of corrosion pits in iron water pipes cannot be due to differences in the concentration of salts in the main water stream and in the pit, as the corrosion products have an alkaline reaction. A probable reason for the formation of the pits is the perforation of the protective coating of rust inside the tube owing to the presence of chlorides or other impurities, followed by occlusion in the rust of the hydrogen set free by the action of the exposed metal on the water and the setting up of an electrolytic cell, hydrogen–electrolyte–iron, which causes rapid corrosion of the iron locally. A. R. POWELL.

Flow in a low-carbon steel at various temperatures. H. J. FRENCH and W. A. TUCKER (U.S. Bur. Standards Tech. Papers, 1925, 19, [296], 619—640).—Upon the application of a fixed load at constant temperature steel elongates for an appreciable time and if the load is not too great this "initial flow" ceases, or is materially reduced, indicating a strain hardening of the material. A "secondary flow" of much less rapidity than the "initial flow" continues under a constant load of suitable magnitude and this in turn is followed by a final rapid flow immediately preceding the fracture of the metal. The relation of the life of the steel to the initial flow appears to be hyperbolic, but though the initial and final stages of flow play an important part in the failure of the metal, the greatest part of the total elongation is produced during the second interval. The importance of the determination of the loads resulting in a zero flow rate during the second stage is emphasised, as not only must the metal be able to resist fracture for a stated time (usually an infinitely long period), but it must not deform appreciably during that period. Comparison of the average flow rate in the second period with the stress–strain relations in short-time tension tests on 0·24% C steel at 295° and 430° showed that the load-carrying ability is well above the proportional limit of the short-time test at 295° and is practically equal to the latter at 430°. The principal factor governing the maximum allowable stress varies with temperature and the type of service, and generally the maximum allowable load closely approximates to the proportional limit of short-time tests at corresponding temperatures. C. A. KING.

Comparative cold-rolling tests of open-hearth steel strip [deep drawing stock] and electrolytic iron strip. J. R. FREEMAN, JUN., and R. D. FRANCE (U.S. Bur. Standards Tech. Papers, 1925, 19, [288], 297—313).—Mechanical tests on cold-rolled electrolytic iron and open-hearth steel suitable for cold drawing showed that electrolytic iron was slightly superior to the steel. Electrolytic iron did not harden so readily and did not increase in tensile strength so rapidly with cold rolling as open-hearth steel. The hardness of both materials increased to a maximum value and then decreased on further rolling. C. A. KING.

Comparative slow bend and impact notched-bar tests on some metals. S. N. PETRENKO (U.S. Bur. Standards Tech. Papers, 1925, 19, [289], 315—346).—Tests made on steels and non-ferrous alloys in an Izod pendulum-type impact machine and a Humfrey slow-bend machine show that the slow-bend test gives values which are related to the tensile yield point and strength of the materials, but the test itself is less convenient in routine practice than the impact test. The notched-bar test is very sensitive in revealing directional differences in the properties of rolled metals, and variations in the individual values in many cases may be accounted for by the structural condition of the metal. The "sharp," "standard," and "round" notches place materials in the same relative order for their resistance to impact or to slow bend, and for brittle metals the sharp and standard notches are preferred. The "deep" notch is considered to be unsatisfactory. C. A. KING.

Permanent magnets. F. STÄBLEIN (Physikal. Z., 1925, 26, 700—707).—The cobalt steels, Koerzit I. (10% Co), Koerzit II. (20% Co), Koerzit III. (30% Co), and a cobalt–molybdenum steel, Koerzit A (15% Co), all manufactured by Krupp, possess a much greater coercive force than, and practically the same remanence as the chromium and tungsten steels previously used for permanent magnets. For Koerzit I., II., and III., the coercive forces are 100, 160, and 220 gauss respectively. A modification of the Koepsel–Kath permeameter, using a shorter (10 cm.) test-piece, is described. Steel for permanent magnets is, in general, sufficiently characterised by the three quantities, remanence, coercive force, and the maximum value of the product $B \times H$ in the left upper quadrant of the hysteresis curve. In considering choice of steel for magnets of different shape, the hysteresis curve needs correction for the self-demagnetisation effect. Where the demagnetisation is small, *e.g.*, in the field magnets of a galvanometer, a high remanence is desirable, and a chromium or tungsten steel is chosen, but for short, straight magnets, *e.g.*, compass needles, or for apparatus subjected to considerable vibration, *e.g.*, magnetos, high coercive force is necessary and here the cobalt steels are to be recommended. A. B. MANNING.

Modern developments in steels resistant to corrosion. W. H. HATFIELD (Engineering, 1925, 120, 657—660).—The resistance of nickel to the action of 10% sulphuric acid and of chromium to nitric acid is reflected in the behaviour of steels containing these elements as constituents, and for a given chromium content, nickel increases the resistance of a steel to hydrochloric and sulphuric acids. Steels containing 12—14% Cr are used in making stainless cutlery, turbine

blading, etc., but do not lend themselves readily to cold working. A steel known as "staybrite," containing 18% Cr and 8% Ni, proved resistant to corrosion by a large number of reagents which attacked steel containing 14% Cr, and possessed greater ductility than the plain, low-carbon, chromium steels. When heated and quenched staybrite does not become hardened but is rendered more ductile. The mechanical properties of this steel in a softened condition were : yield point 15·1 tons/in.^{2}, maximum stress 48·6, elongation 62·8%, reduction in area 45%, hardness (Brinell) 160. The coefficient of expansion (0·000017) was notably greater than that of "stainless" steels and the alloy was non-magnetic. The results of a large number of corrosion tests on iron alloys are tabulated. C. A. King.

Comparison of a new ferro-nickel [permax] of remarkable magnetic properties with permalloy. H. Tscherning (J. Phys. Radium, 1925, **11**, 300—304 ; cf. Yensen, B., 1925, 320 ; Deloraine, 1925, 593).—Permax (an alloy of French manufacture) has little hysteresis in high fields, and considerable hysteresis in small fields. The permeability is high for fields of 1 or 2 gauss, and only moderate in high fields. The alloy has a marked magnetic viscosity in weak fields. Although it lacks the remarkable properties of permalloy it is less susceptible to loss of its characteristic properties. R. A. Morton.

Relations between artificial ferronickel and a meteoric iron containing nickel. M. Peschard (Compt. rend., 1925, **181**, 854—855).—Thermal and magnetic experiments are described which indicate that meteorites are in physico-chemical equilibrium, whereas artificial ferronickels are in a metastable state. S. K. Tweedy.

Special nickel brasses. O. Smalley (Trans. Amer. Inst. Min. Met. Eng., Oct., 1925. Advance copy. 35 pp.).—The addition of nickel in amounts exceeding 1% results in a general improvement in the mechanical properties of brass (59% Cu) in the cast state. Increasing the nickel to 4%—the copper remaining constant—reduces both strength and ductility. The hardness diminishes with increasing nickel content. In amounts up to 4% nickel does not affect the hot-working qualities of $\alpha\beta$-brasses and has a beneficial influence on the shock-resisting properties. The grain size is rendered finer and the quantity of β-constituent is reduced owing to the copper-replacing value of nickel, which is 1·30. Aluminium increases the yield point and ultimate strength of 59 : 41 brass, the limit of rapid hardening being about 1·35%, although it is not until 3% of aluminium has been added that the maximum strength is reached. When this amount is exceeded the strength falls and the alloy loses its ductility owing to the presence of the γ-constituent. The range of heat fragility in β-brass of the copper–aluminium–zinc series is approximately from 226° to 558°. Tin up to 0·5% increases the yield point and ultimate strength without affecting the elongation, reduction of area, or alternating impact strength. Increasing this element to 1·0% hardens without strengthening and impairs the ductility. Iron up to 1% improves the tensile and shock-resisting properties of cast 59 : 41 brass, further additions reducing the latter without increasing the strength. Iron up to 2·52% results in an all-round improvement after hot working, and even in small quantities has a marked refining effect on the structure. Contrary to the findings of Guillet, iron does not act as a zinc-replacing element. The substitution of 3% Ni for 3% Zn in a 1·5% Al 59% Cu brass resulted in general improvement, but reducing the copper to 56% yielded properties which could be equalled by an ordinary aluminimum β-brass. When the copper is increased to 60% and aluminium to 3% the nickel brings about a reduction of strength without affecting the ductility. When forged this alloy gives a higher yield point, ductility, and impact strength. 1% of iron improves the general properties of 59% Cu brass containing 3% Al, but in greater amounts causes a reduction in ductility and impact strength. Iron up to 1% improves slightly the general mechanical properties of brasses containing 1·5% Al with 3% Ni and 3% Al with 3% Ni. Small amounts of tin have a deleterious influence on the properties of a 59% Cu brass containing 3% Ni and 3% Al in the cast condition. Forging breaks down the thin boundaries of the α-constituent around the crystals and produces a closer-grained structure which notably improves the properties of this alloy. The casting temperature of these brasses ranges between 930° and 1030°, depending on composition, and 10% superheat is considered a satisfactory casting temperature ; for heavy ingots this may be reduced to 7%. M. Cook.

Boron in aluminium and its alloys. P. Haenni (Compt. rend., 1925, **181**, 864—866).—Aluminium–boron alloys containing up to 18% B were prepared by heating molten aluminium for periods up to 4 hrs. at 1400° in a crucible lined with boron ; the alloys were cast in heated metal moulds. In the equilibrium diagram for alloys of low boron content the liquidus curve falls continuously down to 620°, corresponding with 8·5% B ; a eutectic line appears at 565° between 1·7% and 8·5% B. The breaking stress and hardness of aluminium are increased by the addition of boron and the elongation under strain is decreased. Addition of boron to aluminium–silicon alloys has a refining effect similar to that produced by sodium and alkali fluorides. S. K. Tweedy.

Aluminium–silicon alloys. A. Petit (Compt. rend., 1925, **181**, 718—719).—The influence of various metals and alloys on the physical properties of aluminium–silicon alloys is recorded. Refining of the alloy is best carried out by squirting in sodium (0·5%) at 775° and casting at 675°. The influence of sodium up to 1% on the properties decreases with increasing rate of cooling after casting. The presence of iron must always be avoided. Copper, magnesium, and alloys of the latter with copper and zinc, present to the extent of 2 to 5%, have the greatest influence. In such cases annealing for an hour at 400° reduces the hardness equivalent to quenching at 500°, and ageing at ordinary temperatures produces a hardening owing to the separation of Mg_2Si or

Al_2Cu, maximum hardness being attained after 70 hrs.; this time is shortened if the temperature is raised to 100°, but above 125—150° a softening occurs. S. K. Tweedy.

Amorphous cement and the formation of ferrite in the light of *X*-ray evidence. F. B. Foley (Trans. Amer. Inst. Min. Met. Eng., Oct., 1925. Advance copy, 8 pp.).—In the freezing of a metal the formation of crystals takes place atom by atom and not cube by cube. This conception is developed to show that at the grain boundaries no amorphous cement exists. The intercrystalline nature of fracture at elevated temperatures is due to the weakness in the structure at the boundaries. Atoms at the boundaries of adjacent grains are only loosely held, having become detached from the lattice of the crystal being consumed in the process of crystal growth and not having finally settled into the lattice of the growing crystal. Slip interference is considered a satisfactory explanation of the raising of the elastic limit by cold working. In the separation of ferrite from austenite it is maintained that the ferrite is not formed within the crystals and rejected to the boundaries, but forms at the exterior of the austenite crystals. Atoms at the crystal boundaries are freer to assume new orientations and possess energy in excess of that necessary for maintaining their positions in the lattice and thus the formation of the α-modification is facilitated at the boundaries. As the γ-lattice progressively breaks down through the addition of atoms to the α-nuclei the carbon atoms are driven towards the centre of the crystal. In the Widmanstättan structure ferrite is found in what were the 111 planes of the austenite. This is partly explained by the predominance of iron atoms at the nuclei of recrystallisation. Since the carbon atom occupies a position between corner atoms along the cube edge any plane parallel to the cube face (001) contains carbon atoms, but none will be found in the octahedral plane. M. Cook.

Elastic properties of alloys: variation with composition. P. Chevenard and A. Portevin (Compt. rend., 1925, **181**, 780—782).—Curves are given for the elastic properties of annealed carbon steels and gold-silver alloys. The modulus of elasticity (μ) varies almost linearly with composition, both in alloys of two structural constituents and also in solid solutions, this being confirmed for alloys of copper with zinc, aluminium, or nickel. On the other hand the elastic contraction is always much less than that of the pure metals, the contraction (δ)–composition curves sinking rapidly from the pure metals to a very flat minimum. The curves for the thermo-elastic coefficient $1/\mu.(d\mu/d\theta)$ pass smoothly but not linearly from one component to the other. W. Hume-Rothery.

Influence of cold-working and quenching on the elastic properties of various metals and alloys.—A. Portevin and P. Chevenard (Compt. rend., 1925, **181**, 716—718).—The influence of thermal and mechanical treatment on the elastic properties of pure metals and alloys was investigated by the method previously described (cf. J., 1918, 91 a). In the case of cold-drawn gold wires the relative torsion modulus diminishes with rising temperature; its value at 15° passes through a maximum at an annealing temperature of about 225°. The thermoelastic coefficient varies similarly with rising annealing temperature; the change in internal friction attains a minimum with an annealing temperature of 250°, and rises only slightly for higher annealing temperatures. Slightly different absolute values are obtained, depending on the time elapsing between annealing and the experimental observations. Qualitatively similar results are obtained with normal solid solutions (*e.g.*, silver–gold alloys); in the case of "anomalous" solutions which have been hardened by quenching or cold-working, such as reversible ferro-nickel alloys, the above effects are observed superposed by the effects of the reversible anomaly modified by cold-working. Such treatment lowers the Curie point and modifies the range of the thermoelastic anomaly. The minimum annealing temperature of ferro-nickels is about 550°. In the case of carbon steel, quenching diminishes the torsion modulus, and increases the thermoelastic coefficient and the change in internal friction, the latter more especially near the eutectic point. The temperature curves of the latter property show a singularity at about 200°, which is independent of the magnetic transformation of cementite and cannot be due to a new polymorphic transformation of iron. S. K. Tweedy.

Leaching mixed copper ores with ferric sulphate. G. D. Van Arsdale (Trans. Amer. Inst. Min. Met. Eng., Feb., 1926. Advance copy. 17 pp.).—The ore treated contains chalcocite and chrysocolla as principal sulphide and non-sulphide constituents respectively, averages 1·25% Cu, and when ready for leaching the limits are approximately 0·1 and 0·9% of copper as sulphide and 0·25 and 1·1% as oxide. After coarse crushing to 1½ in. the ore is further crushed to pass 3-mesh and conveyed to the leaching tanks, where it is in contact with acid ferric sulphate leach liquor for nine days. The liquid is applied on the counter-current principle. Washing consists of the application of six washes in succession and occupies three days. The strongest wash solution is directed into the copper solution. The limits of constituents in the solution for electrolysis are, copper as sulphate 2·5 to 3·5%, total iron 1·5 to 2·5%, ferric iron up to 1·0% or somewhat higher, and free acid from 3·5 to a maximum of 7·5%. The success of the method turns upon the facts that active and efficient ferric sulphate solvent can be regenerated in a non-diaphragm cell by electrolysis with a reasonable yield of copper and that this solvent under proper leaching conditions will give a satisfactory sulphide extraction. Both lead and graphite anodes have been used. Graphite gave a low voltage, a high anode efficiency of conversion of ferrous into ferric iron, and durability in the presence of sufficient ferrous iron, whilst lead yielded higher voltages, a lower anode efficiency corresponding with less ferric iron at the anode per unit of copper deposited, and was durable under the test conditions. A current density of about 15 amp./

sq. ft. is sufficient to counteract the solvent action at the cathode by the ferric iron produced. The average total extraction over a test period of ten months was 77·90%. The tank-house data for the same period averaged 15·00 amp./sq. ft. current density, 62·1% cathode efficiency, 53·9% anode efficiency, and 0·695 lb. of copper per kw.-hr. The copper produced is sufficiently pure to meet standard specifications.
M. COOK.

Determination of small quantities of foreign elements in [scheelite, tungstic acid, and metallic] tungsten. K. AGTE, H. BECKER-ROSE, and G. HEYNE (Z. angew. Chem., 1925, **38**, 1121—1129).—For the determination of the minor constituents of scheelite, 25 g. are fused with 175 g. of sodium potassium carbonate and the insoluble residue is dissolved in hydrochloric acid with a small amount of tartaric acid to retain any tungstic acid in solution. The solution is treated with hydrogen sulphide to remove the heavy metals, the filtrate boiled with excess of hydrochloric acid to remove tungstic acid, and evaporated to dryness, the residue treated to destroy tartaric acid and extracted with water, and the remainder of the analysis conducted in the usual way. For the detection and determination of the rare earths, silica, alumina, magnesia, lime, and the alkalis in tungstic acid or tungsten powder the sample is first roasted in the air, then heated to 400° for 5—20 hrs. in a current of chlorine and sulphur chloride, whereby the tungsten is completely volatilised as an oxychloride leaving the other metals in the residue, which may be analysed in the usual way. Iron is determined by dissolving tungstic acid in ammonia or metallic tungsten in nitric and hydrofluoric acids followed by addition of ammonia and precipitation by hydrogen sulphide; the iron is then determined iodometrically. Zinc is separated from tungsten by means of hydrogen sulphide in dilute acetic acid solution and, after purification, the zinc sulphide is converted into pyrophosphate for weighing. Phosphorus and arsenic are determined together as the magnesium pyro-salts after precipitation with magnesia mixture from the ammoniacal tungstate solution; arsenic is then separated from the phosphorus by distillation with methyl alcohol, hydrochloric acid, and pyrogallol and subsequently determined colorimetrically as sulphide. For the determination of tin the alkaline solution containing tungsten and tin is treated with 5 g. of sodium sulphide and 1 g. of sodium hydrogen sulphate for every 1 g. of tungstic acid present and electrolysed for 4 hrs. at 60° with 1·2 amp. at 4 volts; the precipitated tin is dissolved and determined iodometrically. Bismuth, copper, and lead are separated from tungsten by means of hydrogen sulphide in a chloride solution containing tartaric acid, manganese, nickel, and cobalt by means of hydrogen sulphide in ammoniacal solution, and nickel alone by dimethylglyoxime in the usual way. Molybdenum is best determined colorimetrically in the alkaline tungstate-molybdate solution by addition of xanthate, and vanadium by addition of hydrogen peroxide to the solution after boiling with sulphuric acid to remove the tungsten. Carbon is determined in tungsten metal by combustion in oxygen at 1000—1100°, and oxygen by heating in hydrogen at 1200° for 1—2 hrs. Nitrogen may be determined gasometrically in tungsten metal by dissolving the metal in alkaline ferricyanide solution and measuring the nitrogen evolved. Sulphur is determined by heating the metal in oxygen at 1100°, passing the gases through hydrogen peroxide solution, and precipitating the sulphuric acid so formed with barium chloride, or by titration with thiosulphate of the iodine liberated when a mixture of iodide and iodate is added. A. R. POWELL.

Determination of the grain-size of tungsten powder. K. AGTE, H. SCHÖNBORN, and K. SCHRÖTER (Z. tech. Phys., 1925, **6**, 293—296; Chem. Zentr., 1925, **96**, II, 2013)—Rough values for the grain size of tungsten powder may be obtained by measuring the volume and determining the apparent sp. gr., and more exact values by measuring its adsorptive power for dyestuffs (which is proportional to its surface) by determining the rate of solution in chemical reagents, by calculating its rate of fall in water, or by actual measurement of the grains after embedding a portion of the powder in a soft metal, such as copper. Constant values could not be obtained by measuring the adsorption of methylene-blue from water owing to slight oxidation of the powder. As a measure of the rate of solution of the powder it may be shaken with aqueous ammonia and oxygen, and the oxygen consumption measured. In the fall method it is assumed that the particles are fine enough to fall through water at a constant rate and are almost spherical in shape. A. R. POWELL.

Agglomeration of ores. A. COUSIN (Rev. Mét., 1925, **22**, 697—702).—Sintering a mixture of a finely-divided mineral and a combustible substance by means of a flame impinging on the surface of a layer of the mixture is not a satisfactory method of agglomerating mineral particles owing to the large proportion which remains in fine form. Briquettes made with the aid of bonding material are not strong enough to withstand after-treatment and have the disadvantage of a reduced mineral content. Whilst the sintering of fine minerals in a rotary furnace results in a granular product of sufficient hardness, the low porosity of the grains renders the subsequent reduction of the mineral difficult. The most satisfactory method of treatment is to briquette the mineral without the addition of any bond and to pass the briquettes on cars through a tunnel kiln. Such a kiln at Seraing is 70 m. in length and 2 m. in width, and will treat 140 metric tons of roasted pyrites daily, with a consumption of 60 kg. of carbon per ton of briquettes. The material may be sintered to a sufficient degree to bear the blast-furnace load in subsequent treatment while still retaining the necessary porosity (30—40%) for efficient reduction. It is necessary to study the best briquetting conditions for each type of mineral.
C. A. KING.

Agglomeration of finely-divided ores by the Dwight and Lloyd method. Schneider et Cie. (Rev. Mét., 1925, **22**, 703—710).—The method of agglomerating ores in powder form by heating briquettes of the ore, bonded with clay, in a tunnel furnace has been replaced by the Dwight and Lloyd process of sintering a layer of the ore. A mixture of 56% of ore, 6·5% of pulverised coke, and 37·5% of material which has passed through the process previously is caused to travel on a continuous trough conveyor under the flames from a battery of 10 Méker burners fed with coke-oven gas. The normal thickness of the ore layer is 20 cm. After sintering, the material passes over a screen and grains less than 20 mm. diam., amounting to about 40% of the total, are returned to a further charge. The consumption of fuel is 8 cub. m. of coke-oven gas or in the case of oil firing, 4—5 kg. of oil per metric ton of agglomerated fines. The process is considered to possess advantages in that the product is homogeneous in size and porosity, preliminary drying of the material is not necessary, and desulphurising is satisfactory. C. A. King.

Metallurgy of quicksilver [mercury]. L. H. Duschak and C. N. Schuette (U.S. Bur. Mines, Bull. 222, 1925, 173 pp.).—The metallurgy of mercury has not changed in its main principles since mediæval times, direct furnace treatment of even very low-grade ore being still the most economical process of recovering the metal. Preliminary concentration of cinnabar by flotation or table treatment or a combination of both gives a recovery of 5—20% less mercury than direct furnace treatment and the cost is not appreciably less ; hydrometallurgical processes have the same disadvantages. Recent advances in the metallurgy of mercury have therefore been confined to improvements in the drying and preliminary treatment and improvements in furnace design and in the efficiency of the condensers. Preliminary drying of ore, especially where waste heat is available, is advantageous in that the thermal efficiency of the furnace and condensers is improved and difficulties due to the collection of large amounts of water in the condensers are eliminated. A detailed description with diagrams, illustrations, and cost data is given of the Scott and Spirek furnaces and of several mechanical roasting furnaces. Whatever furnace is used it is essential that complete combustion of the fuel is effected, and that there is always a reasonable excess of oxygen in the furnace atmosphere in order to reduce to a minimum the quantity of mercurial soot, due chiefly to the presence of unroasted mercuric sulphide in the flue gases. Although stoneware pipes are employed as condensers in Europe, American mines used wooden chambers lined with white glazed tiles or simply a series of brick chambers in which the flue gases are cooled progressively from 300° to 40°. The disadvantage of brick chambers is that a certain amount of mercury penetrates the brick floor and a further quantity is absorbed by the walls. Various condenser systems are discussed in detail, and a brief account of the effect of mercury on the health of the workers is given. A. R. Powell.

Patents.

Heat treatment of manganese steel. E. C. R. Marks. From Amer. Manganese Steel Co. (E.P. 242,322, 3.7.24).—Manganese steel castings of the kind described in E.P. 206,183 (cf. B., 1924, 20) are introduced into a furnace immediately after the removal of a previous charge. The furnace temperature will be about 705° at this time, and is allowed to fall to about 580°, from which temperature the furnace is reheated to an upper limit of 1025° until the requisite heat treatment has been effected. C. A. King.

Heat treatment of high-speed steel for the manufacture of permanent magnets. E. C. R. Marks. From Glockenstahlwerke A.-G. vorm. R. Lindenberg (E.P. 242,421, 23.10.24).—High-speed steels to be used for permanent magnets are heated to a temperature above that to which certain kinds of steel have to be heated in order to lower the Ar1 point on cooling the steel, and are then hardened in oil, petroleum, or other mild hardening medium. The steel may or may not contain cobalt. A suitable composition is C 0·6—0·8%, Mn 0·5%, Si 0·25%, Cr 4—5%, Mo 7—8%, Co 1—2%, V 0·5%. C. A. King.

Magnetic structures and method of manufacture thereof. Western Electric Co., Ltd. From Western Electric Co., Inc. (E.P. 242,384, 4.9.24. Addn. to 188,688).—Magnetic structures, *e.g.*, cores, of an alloy containing Ni 78·5% and Fe 21·5% are built up from strips of small cross-section, spaced apart by a phenol condensation product. Such a structure may be assembled with a combustible material as the spacing agent, which is destroyed during the heat treatment to develop high permeability. The structure is then impregnated with a phenol condensation product, and may be heated to a temperature below 200° to form a hard, infusible mass in which the nickel–iron alloy is embedded, and which can be subjected to ordinary usage without impairing the permeability of embedded alloy due to strain. C. A. King.

Refining steel. J. N. Kilby and A. H. Spalton (E.P. 242,475, 6.5.24 and 3.2.25).—Molten steel from a furnace is poured into a container lined with a basic or neutral refining medium, and a cover of the same or similar material is applied so that the molten steel in the container is completely enveloped. The basic lining may consist of a mixture of magnesite 75%, dolomite 25%. An additional refining medium added in small quantity during the pouring of the steel may contain fluorspar 2 pts., lime 2 pts., silica 1 pt., borax glass 1 pt. C. A. King.

Purifying molten metal [steel]. R. Mattice (U.S.P. 1,559,342, 27.10.25. Appl., 21.8.25).—A cylindrical container is filled at one end with a mixture of lime and borax, and at the other with a block of aluminium, and is immersed in molten steel. The fusible plug at the end containing the borax and lime melts, as also does the aluminium block,

and the steel is slowly purified without the explosive reaction which occurs if powdered aluminium is added directly to it. T. S. WHEELER.

Apparatus for the production of electrolytic iron. SIEMENS U. HALSKE A.-G., Assees. of E. DUHME (G.P. 416,082, 23.6.23).—An electrolytic apparatus for the production of pure iron is provided with magnets below the bath or immersed in the electrolyte, and so arranged that the strength of the field decreases towards the cathode. In this way particles that fall from the anode are prevented from becoming attached to the cathode and thus causing the formation of nodules. A. R. POWELL.

Desulphurising agent for iron and other metals. C. EHRENBERG, H. WIEDERHOLD, C. KRUG, M. G. HOLSBOER, K. FISCHER, and STUDIEN-GES. FÜR AUSBAU DER INDUSTRIE M. B. H. (G.P. 417,448, 28.9.23).—A desulphurising agent for iron consists of the alkali or alkaline-earth salts of humic acids. The salts may be carbonised before use, and are preferably added during the smelting of the ore. A. R. POWELL.

Roasting iron ores. A. APOLD and H. FLEISSNER (F.P. 589,472, 21.11.24. Conv., 22.10.24).—Iron ores mixed with a carbonaceous fuel are roasted with an insufficiency of air, and the carbon monoxide so produced is burnt to obtain the heat required for roasting further quantities of ore. A. R. POWELL.

Treatment of iron ores. H. FLEISSNER (F.P. 589,473, 24.11.24. Conv., 28.7.24).—Iron ores, *e.g.*, spathic iron ore, are roasted in a current of an inert, oxidising, or reducing gas. In this way decomposition is effected at a lower temperature, and by suitable adjustment of the composition of the gas or by alternate treatments with oxidising and reducing gases a product may be obtained which is amenable to magnetic separation. A. R. POWELL.

Bearing metal alloys. J. NEURATH (E.P. 238,895, 21.8.25. Conv., 23.8.24).—By the addition of 0·7—2·5% of arsenic to alloys of lead 65—77%, tin 3—14%, and antimony 10—27%, the bearing qualities are made equal to those of bearing metals of the same ternary system containing a high proportion of tin. The increased hardness and compressibility are ascribed to the formation of a ternary lead–antimony–arsenic eutectic, and of a tin–arsenic compound, probably Sn_3As_2. L. M. CLARK.

Bearing metal alloys of high lead content. T. GOLDSCHMIDT A.-G. (G.P. 417,166, 21.4.20).—Small quantities of phosphorus and iron are added to bearing metal alloys, of which lead is the chief constituent. Phosphorus increases the hardness and resistance to wear, whilst iron reduces the brittleness and makes the alloy tougher. A. R. POWELL.

Bearing metal alloys of high lead content. T. GOLDSCHMIDT (G.P. 417,337, 18.6.21. Addn. to 408,229; cf. E.P. 169,703; J., 1922, 942 A).—Arsenic or a mixture of arsenic and phosphorus is added together with the copper specified in the chief patent. A. R. POWELL.

[Foundry] mould composition. A. PACZ (E.P. 240,808, 8.9.25. Conv., 2.10.24. Not yet accepted).—A material suitable for moulds for casting aluminium–silicon alloys or other metals comprises a mixture of a silicon-containing substance of high conductivity in divided form and a binding material, *e.g.*, 10% of clay. The conductive material may comprise one or more silicides of iron, aluminium, calcium, or other metal, *e.g.*, ferrosilicon with 50—90% Si. Other materials such as sand, carbon, coke, carborundum, graphite, or powdered aluminium or other metal may be added to alter the specific gravity or conductivity or both.

Cerium alloys for igniting purposes. A. KRATKY (E.P. 242,361, 9.8.24).—To cerium alloys containing silicon and boron used for igniting purposes, a quantity of potassium, sodium, zinc, calcium, aluminium, lead, or the like is added whereby the particles removed when the mass is scraped are transformed into molten drops of a glass having pronounced pyrophoric properties. An alloy specified has the approximate composition by weight cerium 80%, potassium 4%, calcium 4%, boron 2%, silicon 10%. Those alloys are particularly effective which have such a composition that the glass formed on scraping is composed of cerium oxide 75—90% and lead silicate 10—25%. (Reference is directed in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 16,210 of 1909.) L. M. CLARK.

Pickling process for metal plates. W. THOMAS and M. HAWES (E.P. 242,506, 13.3.25).—A solution, non-injurious to the throat, lungs, skin, or clothes of the user, has the composition by weight, water 1 pt., commercial sulphuric acid (*d* 1·69) 0·58 pt., sodium chloride 0·25 pt., and zinc 0·20 pt. The sodium chloride and zinc are added to the mixture of water and sulphuric acid ; after 4 days the solution is ready for use. Steel plates are immersed in the mixture for ½ hr. at 40° before tinning. L. M. CLARK.

Treatment of lead minerals. L. F. CLARK (U.S.P. 1,548,351, 4.8.25. Appl., 23.4.24).—Ores are treated with alkali dichromate dissolved in copper sulphate solution or with a solution of copper dichromate so as to form a superficial film of lead chromate on the particles of lead minerals present. The ore is then treated with an organic substance (aniline or an aniline acid salt, linseed oil, turpentine, pine oil, etc.), which is oxidised by the lead chromate, and the coated lead mineral is thus made amenable to flotation. Alternatively, a coating of copper cyanide or carbonate may be produced on the particles of lead minerals and the coating treated with acetylene to form copper acetylide therein. C. A. KING.

Metal stock [for use as deoxidising agent]. A. M. HUNT, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,555,978, 6.10.25. Appl., 26.8.20).—Powdered magnesium (10—20 pts.) is mixed with ferrosilicon or ferromanganese (90—80 pts.) and the mixture highly compressed to form a stable

briquette, which is applied to the deoxidation and purification of molten iron or steel. A similar nickel-magnesium briquette is used to purify molten nickel. T. S. WHEELER.

[Alloy for] resisting oxidation at high temperatures. R. F. FLINTERMANN (U.S.P. 1,556,776, 13.10.25. Appl., 14.2.21).—The alloy contains 20—60% of nickel or cobalt, 3—9% of silicon, and the remainder principally iron. T. H. BURNHAM.

Nickel-chromium alloy. W. F. COCHRANE, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,557,025, 13.10.25. Appl., 17.7.24).—The alloy contains 1—5% of chromium, 20—40% of nickel, and the balance principally copper. T. H. BURNHAM.

Nickel-copper alloy. W. F. GRAHAM, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,557,044, 13.10.25. Appl., 28.8.23).—The alloy consists of 30—35% of nickel, 3—7% of iron, and the remainder copper, and contains no lead or zinc. T. H. BURNHAM.

Gold alloy. V. D. DAVIGNON (U.S.P. 1,557,431, 13.10.25. Appl., 4.9.25).—A malleable and ductile gold-copper alloy contains not less than 25% of gold and at least 1% of aluminium. T. H. BURNHAM.

Alloy. F. W. KARITZKY, Assr. to H. B. NEWHALL (U.S.P. 1,559,620, 3.11.25. Appl., 31.12.24).—The alloy contains Zn 85%, Al 10%, and Sn 5%. C. A. KING.

Apparatus for treating ores or the like. G. H. CLEVENGER, Assr. to RESEARCH CORP. (U.S.P. 1,558,965, 27.10.25. Appl., 27.12.20).—The furnace, which is horizontal, consists of a reaction unit, a preheating unit at a somewhat higher level, and a material-transfer connexion between the two units. The charge is passed successively through the preheating and reaction units while a combustible gas is passed in the opposite direction and progressively burned in the reaction unit. The combustion of unburnt gases is effected in a combustion chamber connecting the two units. M. COOK.

Extraction of copper from matte. A. H. HENDERSON (U.S.P. 1,560,574, 10.11.25. Appl., 9.5.21. Renewed 21.4.25).—Copper matte is decomposed by heating it with an alkali carbonate, a fluxing agent, and an alkali hypochlorite. A. R. POWELL.

[Aluminium] alloy. M. E. PAGE (U.S.P. 1,560,845, 10.11.25. Appl., 22.4.25).—An alloy comprising 93·20% Al, 4·55% Au, and 2·25% Cu is claimed. A. R. POWELL.

Preparation of aluminium alloys. A. NEGUI (F.P. 589,223-4, 26.1.24).—The constituent of the alloy of highest m.p. is heated to redness without melting and a portion of the aluminium is added; this reduces the surface oxide and causes the mixture to melt owing to the large amount of heat evolved. Further oxidation is prevented by the oxide layer formed. The remainder of the aluminium is then added, followed by any other constituent. Copper-nickel-aluminium and copper-vanadium-aluminium alloys containing magnesium are readily produced by this method from cupro-nickel and cupro-vanadium. A. R. POWELL.

Electrolytic cell [for production of aluminium]. W. HOOPES, Assr. to ALUMINUM Co. OF AMERICA (U.S.P. 1,562,090, 17.11.25. Appl., 8.11.23).—The carbon bottom-lining of an electrolytic cell is provided with a depression forming a receptacle for molten metal (aluminium) together with a tapping-well extending below the surface on which the molten metal rests and a tap-hole leading horizontally from the bottom of the well. On tapping the cell, liquid floating on the molten metal is prevented from reaching the tap-hole before the receptacle is drained of metal. J. S. G. THOMAS.

Coating aluminium with a strongly-adherent film of copper. G. GÖTZ (G.P. 416,422, 16.5.24).—Aluminium articles may be provided with a strongly adhering copper coating by immersing them for a short time in a boiling solution of copper nitrate and then dipping them into a boiling concentrated solution of copper sulphate. A. R. POWELL.

Nickel-plated article. F. M. BECKET, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,561,900, 17.11.25. Appl., 20.3.24).—A nickel-plated article consisting of an iron alloy containing sufficient chromium to render it rustless under atmospheric conditions is claimed. A. R. POWELL.

Separating molybdenum from ores. W. H. RIDEOUT (U.S.P. 1,562,125, 17.11.25. Appl., 13.3.24).—Ores containing molybdenum are pulped and agitated with a flotation agent in the proportion of pulp 1 pt., flotation agent 2 pts. The latter consists of a mixture of water 90 gals., paraffin 10 gals., soda 4 oz., lye 1 lb. C. A. KING.

Compound metal and method of making it. C. P. BYRNES (U.S.P. 1,562,145, 17.11.25. Appl., 1.3.21).—Loose granules of a metal are introduced into a molten metal of lower melting point than the granules and are allowed to settle under the action of gravity during the cooling of the molten metal. C. A. KING.

Producing molybdenum and vanadium alloys. J. J. BOERICKE (U.S.P. 1,562,201, 17.11.25. Appl., 26.5.20. Renewed 25.9.25).—Molybdenum and vanadium alloys are made by reducing a mixture of a molybdenum or vanadium compound with aluminium in the presence of the alloying metal and a large quantity of indifferent flux. A. R. POWELL.

Tempering light alloys. G. L. WILLIAMS (U.S.P. 1,562,269, 17.11.25. Appl., 8.5.22).—The

alloy is made the cathode in a hot solution of a salt of a heavy metal and electrolysis is continued until the surface is coated with the heavy metal; the alloy is then quenched in cold water.
A. R. Powell.

Production of cyanide solutions [from crude cyanide]. K. F. Cooper, Assr. to Amer. Cyanamid Co. (U.S.P. 1,562,295, 17.11.25. Appl., 21.2.23).—Cyanide solutions suitable for the extraction of precious metals from their ores may be obtained by dissolving a crude cyanide containing carbide in water, adding a sulphide to remove the carbide, and aerating the solution with a gas containing oxygen to convert the excess of sulphide into thiocyanate. A. R. Powell.

Refining of precious metal residues. F. Lau (G.P. 415,720, 24.2.24).—Precious metal residues are melted with borax, sodium nitrate, and a large amount of ammonium chloride to obtain a regulus from which the valuable metals may readily be recovered. A. R. Powell.

Ennoblement of metallic surfaces. W. Guertler (G.P. 416,852, 26.4.22).—The metallic surface is covered with a paste containing molybdenum or tungsten and heated first in an oxidising atmosphere to remove any carbon or sulphur in the paste, then to a high temperature in a reducing atmosphere to reduce the tungsten or molybdenum compounds to metal and cause the latter to alloy with the surface layers of the article. In this way nickel, copper, and iron and their alloys may be rendered resistant to chemical attack.
A. R. Powell.

Smelting tin ores. T. Goldschmidt A.-G., Assees. of L. Schertel (G.P. 417,741, 3.5.23).—Tin ores containing valuable constituents, such as silver, are heated under reducing conditions to a temperature below that at which the gangue sinters, and the reduced tin is leached out with hydrochloric acid, with or without chlorine, ferric chloride, or stannic chloride. The insoluble material is then smelted with materials which yield products that will collect the silver, *e.g.*, lead or copper ores.
A. R. Powell.

Alloy for electrical contacts. Bell Telephone Manuf. Co. (Swiss P. 109,929, 20.6.24).—An alloy for electrical contacts is composed of phosphor-bronze and lead, containing, *e.g.*, 4—5·5% Sn, 1—4% Pb, 0·05—0·25% P, and 90·25—94·95% Cu. Contacts made of this alloy are considerably more durable than those made of lead-free bronze.
J. S. G. Thomas.

Treatment, especially melting, of metals. Westfälische Stahlges. Ossenberg u. Co. (Swiss P. 110,000, 25.10.23. Conv., 31.10.22).—The metal is melted under a layer of flux having a low melting point and a high boiling point. The flux may consist of eutectic mixtures of the halides, carbonates, or oxides of the alkali, alkaline-earth, or light metals, and may contain, in addition, a reducing agent. The operation is carried out in an electric resistance furnace, the walls of which are rendered conducting by treating them at a high temperature in a reducing atmosphere in contact with aluminium powder. The metal to be treated may act as an electrode, in which case it is insulated from the walls of the furnace. A. R. Powell.

Softening aluminium-plated iron articles. F. Jordan (E.P. 243,042, 20.6.24).—See U.S.P. 1,552,744; B., 1925, 885.

Treatment of alloys containing copper and iron. H. Pedersen, Assr. to Orkla Grubeaktiebolag (U.S.P. 1,562,472, 24.11.25. Appl., 16.4.24).—See E.P. 239,768; B., 1925, 885.

Method of producing alloys. A. Pacz (U.S.P. 1,562,654, 24.11.25. Appl., 18.3.20).—See E.P. 160,426; J., 1922, 637 A.

Alloy. C. A. Fontane, Assr. to E. Conti (U.S.P. 1,563,079, 24.11.25. Appl., 2.2.24).—See E.P. 211,456; B., 1925, 508.

Applying metallic coatings to porous [non-metallic] bases. K. Ripper, Assr. to F. Pollak (U.S.P. 1,563,793, 1.12.25. Appl., 21.8.23).—See E.P. 201,567; B., 1924, 985.

Tilting [smelting] furnaces. D. F. Campbell and W. S. Gifford, Assees. of Hirsch, Kupfer- u. Messingwerke A.-G. (E.P. 227,124, 2.1.25. Conv., 3.1.24).

Zinc sulphate from iron ores containing zinc sulphide (G.P. 416,104).—See VII.

Electric-arc furnace (U.S.P. 1,561,731).—See XI.

XI.—ELECTROTECHNICS.

High-frequency-induction electric furnace for very high temperatures. G. Ribaud (J. Phys. Radium, 1925, **11**, 295—299; cf. B., 1925, 554).—A detailed account of the author's graphite-porous carbon furnaces. 10 kilowatts gave a maximum temperature of 3000° for 100 c.c. with one furnace, 2500° for 500 c.c., 1800° for 3 litres volume. With 18 kilowatts and 5 litres volume, the maximum temperature was 2000°. R. A. Morton.

Electrical conductivity of magnesia refractories at high temperatures. J. T. Burt-Gerrans and R. S. Kerr (Trans. Roy. Soc. Canada, 1925, **19**, III., 27).—The specific conductivity of new magnesia bricks increases from 2×10^{-5} mho/c.c. at 870° to 5×10^{-5} mho/c.c. at 1020°, falls to 2×10^{-5} mho/c.c. at 1080°, and then steadily increases to 0·27 mho/c.c. at 1550°. Bricks that have been heated above 1400° and then cooled show a decrease in conductivity between 1200° and 1300°, but reach the same value as for new bricks at higher temperatures. J. S. Carter.

Chemical method of reducing the diameter of tungsten wire [filaments]. W. Samter and K. Schröter (Z. techn. Phys., 1925, 6, 305—306; Chem. Zentr., 1925, 96, II., 2078).—It was found possible to reduce the diameter of the finest tungsten filaments obtained by drawing (0·011 mm.) still further by 35—50% by etching for about $\frac{3}{4}$ min. in a bath composed of a molten mixture of sodium nitrite and nitrate. By slower etching a rougher surface is obtained. By the use of filaments of diameter 0·007 mm. so prepared, it is possible to manufacture lamps taking less current than has hitherto been found possible, *e.g.*, lamps which at 2030° take only about 0·040 amp. while burning over a period of 50 hrs. J. S. G. Thomas.

Transformer oils and methods of testing them. I. Musatti and A. Pichetto (Annali Chim. Appl., 1925, 15, 238—265).—Of the various tests which have been devised in order to obtain a measure of the tendency of a transformer oil to form a deposit, the Brown–Boveri test approaches most nearly the conditions in which such oil functions in practice. Neither air nor oxygen is blown through the oil, oxidation occurring by simple contact of the surface of the oil with the air; further, the temperature used is not excessive in relation to the temperatures encountered when the oil is in actual use. This test, however, takes too much time for practical purposes, it requires a considerable quantity of oil, and the surfaces of contact between the air and the copper catalyst used, and between the copper and the oil, are arbitrary. Modifications are suggested to overcome these disadvantages. The results furnished by a number of oils indicate that no relationship exists between the bromine, iodine, and formolite numbers and the tendency to undergo oxidation, but reveal a certain parallelism between the proportions of unsaturated products soluble in liquid sulphur dioxide and of sludge formed during the oxidation test. The formation of sludge is apparently accelerated by the presence of unsaturated aromatic hydrocarbons in appreciable amount.
T. H. Pope.

Coated filaments for thermionic discharge tubes. Gen. Electric Co., and C. J. Smithells (E.P. 242,438, 11.11.24).—The metal core consists of an alloy of platinum or a metal of the platinum group with either 3% of iron or 5% of chromium and is made by melting the constituent metals and then rolling, hammering, and wire-drawing. Alloys containing iron or chromium are harder and stronger than the pure metal and have a higher electrical resistivity. The electron emission per unit area of filaments coated on alloys of platinum and iron is greater than that of filaments coated on alloys previously used. M. E. Nottage.

Electric incandescence lamps. A. S. Cachemaille (E.P. 242,787, 12.11.24).—In the manufacture of gas-filled electric incandescence lamps a mixture of a hydrocarbon "getter," *e.g.*, diphenylamine, *p*-dibromobenzene, or naphthalene, and a gas, *e.g.*, argon or hydrogen, is introduced into the lamp envelope. If desired the envelope may be flushed out with hydrogen prior to filling.
J. S. G. Thomas.

Mercury vapour device. J. Force, Assr. to Gen. Electric Co. (U.S.P. 1,560,936, 10.11.25. Appl., 11.7.22).—Finely-divided tungsten or silica is sealed in the bulb of a mercury vapour lamp of any usual type. When the lamp is run, a thin, transparent layer is produced on the inner surface of the bulb and prevents the deposition of mercury.
T. S. Wheeler.

Coating electrodes [for electric discharge tubes]. J. E. Harris, Assr. to Western Electric Co. (U.S.P. 1,562,164, 17.11.25. Appl., 4.3.20).—A metal electrode, which combines with alkaline-earth compounds to form compounds not reducible to alkaline-earth oxide upon heating in a vacuum, is coated electrolytically with a deposit of an alkaline-earth metal which is convertible, at least in part, to alkaline-earth metal oxide on exposure to air.
J. S. G. Thomas.

Deoxygenation of enclosed atmosphere [in transformers]. C. J. Rodman, Assr. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,557,092, 13.10.25. Appl., 23.12.22).—The space above the surface of the oil in an enclosed transformer is connected by two tubes to a reaction vessel containing a finely divided alloy of magnesium and lead mounted on asbestos. Initially the alloy is electrically heated, and when the air in the enclosed space circulates over the alloy the oxygen is rapidly absorbed. The reaction chamber is connected with the air by a mercury seal and any oxygen which may enter as the transformer "breathes" is slowly absorbed at the ordinary temperature, provided water vapour is present. Sludging of the oil is thus prevented.
T. S. Wheeler.

Electric-arc furnace. F. Kostka, Assr. to Rheinische Metallwaaren- u. Maschinenfabrik (U.S.P. 1,561,731, 17.11.25. Appl., 30.12.24).—The part of the electrode lying in the wall of an electric-arc furnace for melting metals is surrounded by a tube contained within a sleeve of non-conducting material. One end of the sleeve is embedded in the fireproof furnace wall, whilst the other end holds the metallic part of the packing for the electrode at such a distance from the wall of the furnace that short-circuiting is prevented.
J. S. G. Thomas.

Electric furnace. J. Sechowski (U.S.P. 1,562,261, 17.11.25. Appl., 17.8.23).—The casing of an electric furnace is divided internally into separate compartments communicating at the bottom below the vertical dividing partition. The top of the casing is provided, above one of the compartments, with an opening whereby air may be driven through this compartment into the other compartment. Horizontal baffles disposed in staggered relation are arranged in the latter compartment, being mounted alternately on the vertical partition and on one of

the walls of the compartment. The width of the baffles is equal to that of the compartment, whilst their length is less than that of the compartment. Electric heating elements are arranged in transversely spaced rows beneath the baffles.

J. S. G. Thomas.

Electric furnace with electrodes arranged in the region of the furnace hearth. W. Kunze (G.P. 416,736, 22.7.21).—In an electric furnace, auxiliary electrodes equal in number to or many times the number of the principal electrodes are arranged along the circumference or on the floor of the hearth. Current is supplied to the auxiliary electrodes exclusively from a supplementary transformer controlled by the consumption of current in the arc electrodes. A short circuit is arranged in parallel with the primary of this transformer, so that the latter may be thrown out of circuit, leaving the arc heating alone in operation.

J. S. G. Thomas.

Electrical resistors. J. L. Capelle (Swiss P. 109,991, 27.6.23).—Electrical resistors are composed of an electrically-conducting material, *e.g.*, a mixture of carbon and a powdered metal, together with a non-conducting material, *e.g.*, alumina, cement, lime, etc., or mixtures of these substances. The raw materials mixed with water and, if desired, with a binder, are moulded and dried at a temperature not exceeding 500°. The metal–carbon mixture may be arranged either outside the non-conducting material, or between layers of the latter. By the use of mixtures of carbon and metal a resistance which is constant between certain limits of temperature is obtained. J. S. G. Thomas.

Electrolyte for galvanic elements. R. Pörscke (U.S.P. 1,562,517, 24.11.25. Appl., 29.8.21).—See E.P. 190,226; J., 1923, 149 a.

Impermeabilising substances for absorbing gas [for use in electric batteries]. E. A. G. Street, Assr. to Soc. Anon. le Carbone (U.S.P. 1,563,673, 1.12.25. Appl., 30.6.22).—See E.P. 198,656; B., 1924, 986.

Electric discharge devices and means for operating them. Brit. Thomson-Houston Co., Ltd., Assees. of C. G. Found and I. Langmuir (E.P. 240,149, 4.9.25. Conv., 18.9.24).

Magnetic structures (E.P. 242,384).—See X.

Electrolytic cell (U.S.P. 1,562,090).—See X.

Alloy for electrical contacts (Swiss P. 109,929).—See X.

Melting metals (Swiss P. 110,000).—See X.

XII.—FATS; OILS; WAXES.

Oil content of the seeds of the opium poppy. H. E. Annett and M. N. Bose (Mem. Dep. Agric. India, Chem. Series, 1925, **8**, 39—43).—The oil content of the seed of the opium poppy grown in the United Provinces is about 50% on the dry matter of the seed, and variations in the contents of the seeds of nine pure races tested are insignificantly small. Variations in content due to season, climate, manures, the use of terminal or lateral capsules, and lancing the capsule for opium are also negligible.

C. O. Harvey.

Tests for rancidity in fats. J. Bulir (Chem. Listy, 1925, **19**, 357—362).—Rancid fats contain products which give peroxide reactions, and in testing for these *p*-diaminodiphenylamine sulphate is a good reagent. A surer method is as follows: shake 1 c.c. of the fat dissolved in 1 c.c. of light petroleum with 2 c.c. of 20% alcoholic potassium iodide solution, add 15 c.c. of water, shake, and test the aqueous layer with starch paste. A blue colour indicates that the fat is rancid. B. W. Anderson.

Cacao beans and cacao products. Fincke.—See XIX.

Patents.

Purifying oils and fats under a high vacuum by means of steam or the like. Metallbank u. Metallurgische Ges., and W. Gensecke (E.P. 242,739, 17.9.24).—Steam led into the oil or fat contained in a vacuum vessel, is withdrawn from the vessel by a special injector (cf. E.P. 237,309) and may then be passed through a second vacuum vessel before being condensed. The injector is provided, in front of the convergent guide conduit, with an additional nozzle which increases the velocity of the steam and decreases its pressure. The vacuum obtained is higher and the steam consumption lower than in the ordinary vacuum condenser plant.

G. T. Peard.

Mixed esters [glycerides] of lower and higher fatty acids. G. L. Schwartz, Assr. to E. I. du Pont de Nemours and Co. (U.S.P. 1,558,299, 20.10.25. Appl., 11.4.22).—Glycerin is heated with acetic acid and the acids from coconut oil in presence of a small quantity of sulphuric acid, and any water formed is removed by passing a current of nitrogen through the mixture. A product which is for the most part the diacetylmonoglyceride of the coconut oil acids is obtained. It has a low freezing point, is non-volatile, and rapidly colloids pyroxylin. A similar product is obtained by using coconut oil, acetic acid, and glycerin. T. S. Wheeler.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Detection of rosin [in linseed oil]. H. Wolff (Farben-Ztg., 1925, **31**, 515).—The apparently positive reaction given by linseed oils containing no rosin in the Liebermann–Storch test differs from the genuine reaction in that the coloration develops but gradually through stages of brown and red, to blue-violet. The greatest intensity of violet colour attained is, furthermore, only reached after an interval of time corresponding to the change of violet to brown or green in the case of the true rosin reaction. In the presence of rosin the violet

coloration invariably develops immediately. The distinction between the two cases is more marked if the oil under test be previously dissolved in a few drops of light petroleum previous to solution in acetic anhydride. It is recommended that tests be made simultaneously against a control of linseed oil containing 0·5—1·0% of rosin, no violet coloration whatever or development to a maximum intensity of violet with simultaneous fading or disappearance of violet in the control test indicating absence of rosin. A. DE WAELE.

Catalytic effect of lead and manganese on the drying of China wood [tung] oil. G. E. LUDWIG (Ind. Eng. Chem., 1925, 17, 1160—1161).—Varying amounts of litharge or manganese borate were added to mixtures of tung oil and rosin which had been heated to 288° for 12 min., then cooled to 218° (litharge) or 200° (manganese borate); the temperature was maintained for 10 min., after which the oil was cooled to 177°, and mixed with the thinner. Drying tests were made with the product on glass plates, and curves (% metal plotted against time taken to "dry to touch") drawn for the two driers. The lead curve was even and regular, whereas the manganese curve, after the initial fall to a well-marked minimum drying time, shows a rise and then a somewhat irregular flat portion. The use of more than 0·26% of lead does not improve the drying properties very much, and there is no advantage at all in using more than 0·50%. With manganese the most efficient amount was found to be 0·03%, and the drying time increased with further increase in the proportion of metal. The litharge-in-oil samples gave a slight gradation of colour and amount of sedimentation on keeping. Above 0·5% of lead the amount of sediment was excessive. The sedimentation was more marked with manganese. D. WOODROFFE.

"Two-type" lacquer solvents. D. B. KEYES (Ind. Eng. Chem., 1925, 17, 1120—1122).—"Two-type" solvents, *i.e.*, ester-alcohols, ether-alcohols, keto-alcohols, keto-esters, keto-ethers, or ester-ethers, are generally better than "single-type" solvents, *i.e.*, simple esters, alcohols, ethers, or ketones, for use in the manufacture of nitrocellulose and resin lacquers. The methods of preparation and properties of a number of these "two-type" solvents are given, viz.: — Ester-alcohols, ethyl glycollate (b.p. 160°), ethyl lactate (b.p. 152°), ethyl β-hydroxypropionate (b.p. 187°), ethyl citrate (b.p. 261—263°/300 mm.), ethyl malate (b.p. 248—252° decomp.), ethyl tartrate (b.p. 280°), ethyl glycerate (b.p. 230—240°), ethyl salicylate (b.p. 232°), ethylene glycol monoacetate (b.p. 182°). Ether-alcohols; ethylene glycol monoethyl ether (b.p. 134°), trimethylene glycol monoethyl ether (b.p. 160—161°), glycerol diethyl ether (b.p. 191°), glycide (b.p. 161—163°), diethylene glycol (b.p. 250°). Ketone-alcohols: pyruvyl alcohol (acetol) (b.p. 145°), diacetone-alcohol (b.p. 164°), dihydroxyacetone (m.p. 68—75°), acetonyl-methyl alcohol. Ketone-esters: ethyl pyruvate (b.p. 160°), ethyl acetoacetate (b.p. 181°). Ketone-ethers: ethoxyacetone (b.p. 128°), ethoxyethyl methyl ether. Ester-ethers: ethyl glycollate ethyl ether (b.p. 152°), ethyl α-ethoxypropionate (b.p. 155°). L. A. COLES.

Working-up tar oils. KÁRPÁTI.—See III.

PATENTS.

Manufacture of artificial shellac. SOC. OF CHEM. IND. IN BASLE (E.P. 221,205, 21.8.24. Conv., 30.8.23).—Artificial resin obtained by sulphuration of a phenolic compound is subjected to the action of a base, such as *cyclo*hexylamine or ammonia, in the presence of water if necessary. The original artificial resin, if desired, may first be treated with formaldehyde and with agents imparting elasticity. D. F. TWISS.

Composition for lubricating, dissolving and preventing rust and the like. A. DOKTER (E.P. 241,678, 13.9.24).—The composition consists of a mixture containing 7·5 pts. by weight of chemically pure zinc-white, 4·5 of lampblack, 33·0 of American graphite, 7·5 of consistent grease, 7·5 of technically pure horse-fat, and 18·0 pts. of purified seal oil. It forms a non-drying acid-free paste capable of withstanding the action of the strongest acids and not becoming separated at temperatures as high as 255°. It remains elastic at high and at low temperatures. M. COOK.

Production of thermoplastic compositions for use in the manufacture of moulded articles. T. HOUGH (E.P. 241,807, 20.4.25).—By mixing two or more copals, gums, or resins with shellac, *e.g.*, kauri copal 40, dammar 20, hardened rosin 25, and shellac 15 pts., and heating under pressure to 200—350° for 30—60 min., a product is obtained which can be mixed with more shellac, suitable fillers, and colouring matter to produce a material capable of being softened by heat and moulded to shape under pressure. D. F. TWISS.

Priming composition [for varnishes and lacquers containing drying oils]. G. ROTH A.-G., and R. WEITHÖNER (E.P. 242,379, 28.8.24).—Sufficient aluminium hydroxide to combine with the resin is added to a mixture of a resin and a drying oil or a varnish or lacquer containing a drying oil, and the whole is then warmed, triturated, and thinned with a solvent, with or without addition of a drier. Alternatively a basic aluminium salt of a resin or fatty acid may be added direct. Suitable proportions are 2 pts. of aluminium hydroxide, 30 of boiled linseed oil, 35 of oil of turpentine, and 5 of lead-manganese drier. S. BINNING.

Manufacture of linoleum covering material. G. SCHICHT and A. EISENSTEIN (E.P. 242,832, 3.2.25).—The solid raw materials are mixed with the fluid ingredients so as to form a pulverulent mass, which is exposed to atmospheric oxidation at a temperature above 70°. The constituents still lacking are introduced during or after the oxidation process, the addition in the former case providing

a means of preventing any excessive rise in temperature. The method is rapid and safe.
D. F. TWISS.

Synthetic resin coating composition. C. ELLIS (U.S.P. 1,557,519, 13.10.25. Appl., 7.2.21. Renewed 9.1.24).—A mixture of *m*- and *p*-cresol is heated with sulphur chloride and the plastic product is blown with steam at 120° to deodorise it and then baked in thin layers for 6·5 hrs. The product when mixed with an equal weight of boiled linseed oil and thinned with turpentine and amyl acetate gives a varnish which dries to a clear film resistant to acids and alkalis. T. S. WHEELER.

***cyclo*Hexanol–aldehyde resin.** C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,557,521, 13.10.25. Appl., 30.3.23).—*cyclo*Hexanol is heated with 30% of its weight of formaldehyde in presence of 50% of its weight of 50% aqueous caustic potash. A fusible resin is obtained which becomes infusible on heating, especially if hexamethylenetetramine is added. T. S. WHEELER.

Keto-alcohol resin. C. ELLIS (U.S.P. 1,557,571, 20.10.25. Appl., 27.2.22).—Ketobutyl alcohol heated with 1% of its weight of alkali rapidly sets to an infusible resin. T. S. WHEELER.

Decolorising and refining gums and resinous products of coniferous pine trees. A. R. AUTREY (U.S.P. 1,559,399. 27.10.25. Appl., 13.8.24).—Solid gum turpentine is melted in a closed vessel, filtered hot, and stirred with fuller's earth. The decolorised material is then distilled with steam at ordinary or reduced pressure at a temperature not above 160°. The residue in the still is filtered hot and yields a resin of high quality. The distillate is treated with decolorising charcoal and is fractionated to give turpentine oil. T. S. WHEELER.

Composition for preventing rust. (E.P. 241,678).—See XIII.

Acid and salts strongly absorbing ultraviolet rays. (E.P. 242,721).—See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation [of rubber latex] with sodium silicofluoride in conjunction with *p*-nitrophenol. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1925, **7**, 657—658).—Coagulation of standard rubber latex (3000 c.c.) by the addition of a mixture of sodium silicofluoride (2 g.) and *p*-nitrophenol (0·5 g.) gives a product similar to that of coagulation with acetic acid in appearance and rate of vulcanisation, but free from bubbles and tendency to mouldiness. D. F. TWISS.

Calender- and creep-effect in unvulcanised rubber. W. DE VISSER (Diss., Delft, 1925; Gummi-Ztg., 1925, **40**, 457—458, 511—513).—When stressed in the direction of calender grain rubber shows a steady increase in load with increase in stretch; across the grain or in rubber without grain the load remains approximately constant over a considerable increase in extension. Previous heating or prolonged mechanical working reduces the extent of development of grain; subsequent heating also causes the effect to disappear. Rubber sheet with calender grain is doubly refractive and dichroic, and exhibits a distinct Debye–Scherrer diagram; its specific gravity is higher than normal, and it tends to become hard and brittle. The degree of creep shows no simple relation to the extent of calender grain; the former is probably explained by Lunn's views (Ann. Repts., 1924, 385), whereas the latter is probably associated with the development of a definite orientation of the rubber particles with perhaps partial crystallisation. Gutta and balata can exhibit marked calender grain; *Castilloa* rubber and *Hevea* rubber are comparable in behaviour but with *Ficus elastica* rubber the effect is only weak. D. F. TWISS.

United States Government master specification No. 59a for rubber goods. (Methods of physical tests and chemical analyses.) (U.S. Bur. Standards Circ. No. 232, 1925, 42 pp.).—Details are given as to the methods of taking samples, of measuring tensile strength, extensibility, permanent set, adhesion, and hardness, and of applying tests as to behaviour when subjected to steam under 75 lb. pressure, to hydrostatic pressure (for hose), and to accelerated ageing at 70°. The chemical tests include determination of specific gravity, acetone extract (with further determination of unsaponifiable and hydrocarbon content), chloroform extract, alcoholic potash extract, free sulphur, total sulphur, ash, barium sulphate, antimony, free carbon, glue (from nitrogen content), and cellulose. Rubber is calculated by difference either by means of the preceding results, or with the aid of an additional determination of the proportion insoluble in mineral oil at 150—155°.
D. F. TWISS.

PATENTS.

Vulcanisation of rubber. S. J. PEACHEY and A. SKIPSEY (E.P. 242,464, 9.12.24).—Rubber solutions treated with insufficient phosphorus sulphide to effect gelling under normal conditions, may be caused to gel at once by the addition of ammonia. Rubber vulcanised by phosphorus sulphide (*cf.* E.P. 230,637; B., 1925, 368), if subjected to an after-treatment with ammonia, is improved in its mechanical characteristics. D. F. TWISS.

Treating rubber and similar materials. T. WHITTELSEY and C. E. BRADLEY, Assrs. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,559,393, 27.10.25. Appl., 19.2.20).—Three mols. of aniline are dissolved in benzene and treated with 1 mol. of sulphur chloride. Aniline hydrochloride is precipitated, and on evaporation phenyliminothiosulphurous acid, NPh : S : S, is obtained. This substance has the property of rapidly vulcanising rubber, and may be used alone or mixed with other vulcanisers and accelerators. Other amines give similar products; for example, ammonia and sulphur chloride yield a substance with the formula, $S:S(:N\cdot S:N)_2$, having similar properties. T. S. WHEELER.

Treating rubber. E. B. Spear, Assr. to Goodyear Tire and Rubber Co. (U.S.P. 1,560,488, 3.11.25. Appl., 6.1.23).—Unactivated carbon saturated with ammonia and a second batch saturated with hydrogen sulphide, formaldehyde, or carbon dioxide are mixed with rubber. The substances with which the carbon is saturated interact and the products act on the rubber in the usual manner. Any gas used to treat rubber may be added to it in this manner in order to obtain more intimate contact.
T. S. Wheeler.

Accelerator for vulcanisation of rubber. A. Cambron, Assr. to Roessler and Hasslacher Chemical Co. (U.S.P. 1,562,146, 17.11.25. Appl., 27.6.25).—Rubber is vulcanised with the aid of the steam-blown reaction product of an aldehyde and an aromatic amine. D. F. Twiss.

Waterproofing cement etc. (E.P. 242,345).—See IX.

Photographic papers (E.P. 241,769).—See XXI.

XV.—LEATHER; GLUE.

Action of ultra-violet light on hide protein. A. W. Thomas and S. B. Foster (J. Amer. Leather Chem. Assoc., 1925, **20**, 490—494).—Portions of dry hide powder were submitted to the rays from a 1000-candle-power Cooper-Hewitt mercury arc quartz lamp for several weeks. The powder became coloured a bright canary-yellow even when the air was replaced by nitrogen. When the treated powder was treated with an equal weight of water at the ordinary temperature 27·8% dissolved. Portions of treated and untreated powders were shaken with solutions of hemlock extract and quinone respectively at different p_H values. The results showed that the capacity of the hide powder to combine with hemlock tannin at p_H 3·8 was greatly reduced by exposure to ultra-violet rays. No difference was noted with the quinone tannage. This shows a marked difference in the nature of vegetable and quinone tannages. If the simple electron emission theory of effect of irradiation were correct, the irradiated collagen should show greater affinity for the vegetable tannin.
D. Woodroffe.

Suggested tests on shoe upper leather. A. Rogers (J. Amer. Leather Chem. Assoc., 1925, **20**, 495—497).—A method of determining resistance to tearing at stitch holes is described. Leathers about which complaints of cracking were received, were invariably undertanned and did not withstand the boiling test. D. Woodroffe.

Tests with various hide powders using a buffer solution of p_H 4·6. J. G. Parker and J. T. Terrell (J. Soc. Leather Trades Chem., 1925, **9**, 479—480).—Samples of solid chestnut extract, oakwood, and sulphited quebracho extracts were respectively analysed with untreated B. 12 hide powder and batches 9, 11, 12, and 13 treated according to the method of the committee of the Society of Leather Trades Chemists for hide powder (cf. J. Soc. Leather Trades Chem., 1925, **9**, 405). Hide powders which gave bad results before treatment were completely altered by treatment and then gave uniform and satisfactory results. The use of a buffer solution of p_H 4·6 for treating hide powders before use in tannin analysis brings them all to the same standard. The differences in the non-tannin results in the above experiments varied from 0·3% to 0·8% with 6 different powders.
D. Woodroffe.

Deliming heavy hides. G. Baggini and L. Chiesa (Bull. Uff. Staz. Sperim. Ind. Pelli, 1925, **3**, 285—288).—Deliming by means of either sodium bisulphite or ammonium chloride or by bating preparations accelerates the subsequent tanning but tends to lessen the tensile strength, and if great strength is required the best results are obtained by the use of hydrochloric acid. The action of ammonium chloride is analogous to that of the bran drench. T. H. Pope.

Determination of acidity in synthetic tannins. G. A. Bravo (Boll. Uff. Staz. Sperim. Ind. Pelli, 1925, **3**, 296—298).—The method proposed by the American Leather Chemists Association (J. Amer. Leather Chem. Assoc., 1922, **17**, 627; J., 1923, 152A) gives (1) for a tannin extract containing 3% of synthetic tannin, acidity as sulphuric acid 1·02% and as benzenesulphonic acid 2·20%; the ordinary method gave 2·15% of sulphuric acid; (2) for a synthetic tannin, acidity as sulphuric acid 15·6%, and as benzenesulphonic acid 8·2%; the ordinary method gave 12·67% of total acidity as acetic acid. The above American method may be accelerated by mixing the liquid containing the gelatin with kaolin and filtering through an alundum crucible. The method serves well with synthetic tannins, but is not nearly so suitable for tanning extracts containing synthetic tannins. T. H. Pope.

Control of chrome liquors in the one-bath chrome tanning process. [Report of committee of the British section of the Society of Leather Trades Chemists.] D. Woodroffe (J. Soc. Leather Trades Chem., 1925, **9**, 480—485).—The basicity of various one-bath chrome tanning liquors has been determined by the Procter-McCandlish method and also by a modified method (cf. J., 1921, 669A). The modified method using sodium chloride showed an apparent increase in the acidity of the liquor. Of two commercial one-bath chrome tanning extracts of different basicities, the more acid product tanned more quickly than the basic product. The basicity of the latter was adjusted to that of the former, but their tanning properties were not made identical. A comparison of the precipitation figures and basicities of a series of one-bath chrome liquors showed that the precipitation figure was affected by the method of manufacture of the liquor. Liquors with the same precipitation figure, but made by different methods, did not tan equally well. The p_H value of these different chrome liquors was determined and it was shown that the more acid

commercial extract had a higher p_H value than the other commercial extract. The p_H value is suggested as a better method of controlling the tanning properties of liquors of the same chromium content than the basicity figure, especially in the case of chrome liquors manufactured by different methods. D. WOODROFFE.

Determination of fat and water-soluble [matter] in leather. A. COLIN-RUSS (J. Soc. Leather Trades Chem., 1925, 9, 455—478).—Experiments have shown that when a leather is immersed in a fat solvent, an equilibrium is attained, and at this point the ratio of the concentration of fat retained by the leather to the concentration of fat in the external solvent is a constant, which is characteristic of the system. The fat content of a leather can be determined without its complete removal if the constant is known. To determine the constant, a known weight (40 g.) of leather is placed in a calibrated vessel, covered with a solvent, the vessel gently shaken to remove air bubbles, and the mixture made up to 100 c.c. with solvent. The vessel is closed, allowed to remain for 24 hrs. at room temperature, the liquid decanted and thoroughly drained off into a measuring vessel, the leather covered with a fresh lot of solvent, and the sequence of operations repeated. The equilibrium constant, K, is given by $E_2.V/(E_1-E_2)m$, where E_1 and E_2 are the amounts of fat removed in the first and second decantations respectively, V is the volume of liquid decanted, and m is the weight of the leather minus the total fat. The total fat content is given by $F=E_1^2/(E_1-E_2)$. The fat content of samples of different chrome- and vegetable-tanned leathers determined by the above method was often in close agreement with that obtained by the usual Soxhlet extraction, but not always. The method is applicable to all tannages and is independent of the state of division. The iodine value of different extracts was determined in one case and successive extracts had higher iodine values. The above method has also been applied to the determination of water-soluble matter in vegetable-tanned leathers by first degreasing weighed strips of the leather and using distilled water as the solvent. The results obtained for a number of different leathers were about 0·7 of the water-soluble matter as determined by the official method. Successive extractions were made on several vegetable-tanned leathers and the logarithm of the total amount extracted from the leather plotted against the number of extractions, and it was shown that the first few points were on a straight line, after which the amount of extract increased unduly. This is attributed to the hydrolysis of the collagen tannate and thus the above new method is only applicable to the determination of water-soluble matter provided the first two extractions are obtained under conditions that exclude hydrolysis. The water-soluble matter as determined by the official method includes solid matter arising from hydrolysis of the collagen tannate and is therefore too high.
D. WOODROFFE.

Working-up tar oils. KÁRPÁTI.—See III.

Mordant for dyeing glove skins. SIMONCINI.—See VI.

XVI.—AGRICULTURE.

Physiological importance of extractable nutrients, a contribution to the determination of the manurial requirement of soils by chemical means.—E. BLANCK and F. ALTEN (J. Landw., 1925, 73, 219—230).—The amount of phosphoric acid extracted from a soil by a variety of acid solvents was determined, the weaker acids being used both with and without making allowance for the carbonates present in the soil. Oats were then grown on sand cultures receiving phosphoric acid (in the form of dicalcium phosphate) equivalent to the varying amounts extracted by the different solvents, each pot having the same application of other essential nutrients. It is concluded that only dilute acids are suitable solvents for determination of assimilable phosphoric acid in soil. Of those tried, 1% nitric acid, 1% acetic acid, and 0·5% citric acid gave satisfactory results, if the amount used was adjusted to allow for neutralisation by the carbonates present. Water saturated with carbon dioxide was, however, quite unsuitable. C. T. GIMINGHAM.

Determination of manurial requirements of soils by Mitscherlich's method. E. A. MITSCHERLICH (Z. Pflanz. Düng., 1925, B4, 473—478).—A reply to the criticisms of Gerlach (cf. B., 1925, 220, 731). C. T. GIMINGHAM.

Acetone method of extracting sulphur from soil. R. H. SIMON and C. J. SCHOLLENBERGER (Soil Sci., 1925, 20, 393—396).—Details are given of a method for the determination of elementary sulphur in soils by extraction with acetone and subsequent oxidation to sulphate. Sulphates present in the soil are not removed by the solvent and in studies of the rate of oxidation of sulphur in soil errors from this source are eliminated and a positive measure is obtained. C. T. GIMINGHAM.

Nitrification in soils. H. N. BATHAM (Soil Sci., 1925, 20, 337—351).—Laboratory studies on the nitrification of some decomposition products of proteins which may occur in the soil are recorded. The most readily nitrifiable of the compounds tested was tryptophane, followed by phenylalanine, α-alanine, leucine, tyrosine, and cystine in the order named, but the differences noted are small and doubtfully significant. All are apparently less readily nitrified than ammonium sulphate. C. T. GIMINGHAM.

Action of zeotokol [powdered dolerite] on plant growth. E. BLANCK and F. ALTEN (J. Landw., 1925, 73, 213—218; cf. B., 1925, 80).—A further series of pot experiments, with oats, on three types of soil, in which zeotokol was applied mixed with the whole of the soil, or with the top layer only, or on the surface, confirms the view that this material has no significant effect on plant growth. C. T. GIMINGHAM.

Development and composition of potato plants under the influence of different manuring. W. HUXDORFF (J. Landw., 1925, 73, 177—212).—Whole plant samples were taken from each of eight differently manured plots of potatoes at six dates during the growing period. The treatments of the plots included manuring with potassium, nitrogen, and phosphorus singly and in all combinations. The yields and percentages of potassium, nitrogen, and phosphorus were determined, the haulm and the tubers being dealt with separately. Numbers of tubers per plant and measurements of leaves and stems were also recorded; and, in addition, sections of leaves and stems were cut and observations made on the effect of the treatments on the structure and size of the cells of various tissues at different stages of growth. On the whole, the results tend to confirm the generally accepted views as to the characteristic effects of the three primary nutrients on plant growth; but, contrary to the conclusions of some previous workers, it was found that, with both nitrogen and potassium, the potato will take up an abnormal amount of whichever of these nutrients is in excess—an amount which may be greater than the actual requirements at the stage of growth reached. The analyses and botanical observations are discussed in detail.

C. T. GIMINGHAM.

Bacterial oxidation of sulphur in pond deposits and its practical importance. H. FISCHER (Zentr. Bakt. u. Parasitenk., Abt. II., 1925, 65, 35—42; Chem. Zentr., 1925, 96, II., 1687).—The autotrophic organisms responsible for the oxidation of sulphur to sulphates in pond deposits are favoured by alkalinity of the medium and inhibited by acidity and the presence of organic matter. Their behaviour is similar to that of nitrifying organisms. Phosphoric acid, which is important for the production of fish, is brought into solution by the action of the sulphate ion on the phosphates in the deposit.

C. T. GIMINGHAM.

Investigations on "tuba" [derris]. B. A. R. GATER (Malay Agric. J., 1925, 8, 313—329).—As an insecticide, *Derris*, to which the name "tuba" is usually applied, is comparable with nicotine. The Malay word "tuba" is not confined to species of *Derris*, but eleven "tuba" plants not *Derris* species were found to have no practical value as insecticides. The *Derris* species, and also varieties of the same species, vary in toxicity. The best plant for insecticidal purposes appears to be the "*Tuba rimba*" variety of *Derris elliptica*. An account of various pests of *Derris* species, and of means of combating them is given.

C. O. HARVEY.

Mechanical analysis of sediments. COUTTS and CROWTHER.—See I.

Size distribution of particles. WERNER.—See I.

Assimilation of carbon dioxide by plants. NELLENSTEYN.—See II.

Influence of manures on yield and morphine content of latex of opium poppy. ANNETT and SINGH.—See XX.

PATENTS.

Manufacture of manures. RHENANIA VEREIN CHEM. FABR. A.-G., and H. BRENEK (E.P. 242,512, 20.3.25).—Mineral phosphates are heated to about 1200° with silica or silicates and an alkali salt, preferably the carbonate or sulphate. The proportions in the mixture are so adjusted that one-third of the lime combined with phosphoric acid in the mineral is converted into calcium silicate and is replaced in the phosphate complex by alkali. Sufficient silica must be added (taking into account that present in the mineral) to convert the whole of the lime not combined with phosphoric acid into calcium silicate. The silica required may be added in the form of mineral phosphate rich in silica. The product contains the phosphate in a readily available condition.

C. T. GIMINGHAM.

Fertiliser. O. REITMAIR (Austr. P. 99,793, 31.5.21; Swed. P. 57,113, 20.5.22).—Crude phosphates containing carbonate are powdered and treated with sufficient of an inorganic acid or solution of an acid salt to convert the carbonate into hydrogen carbonate. If an acid other than sulphuric acid, or an acid salt other than a bisulphate be used to effect the transformation, a soluble sulphate is also added in order that a dry product may be obtained.

J. S. G. THOMAS.

Making sulphur products [fungicides]. W. C. PIVER (U.S.P. 1,559,984, 3.11.25. Appl., 28.1.25).—Casein (2 pts.) is dissolved in water (97·5 pts.) containing sodium hydroxide (1·5 pts.), and the solution is mixed with finely-divided sulphur (200 pts.), the mixture being maintained alkaline throughout. Colloidal dispersion of the sulphur takes place and a product of value as a fungicide is obtained.

T. S. WHEELER.

Preparation of a product for spraying plants. M. BUCHNER and W. BACHMANN (G.P. 416,800, 4.5.20).—Copper–arsenic compounds, in the presence of ammonia, react with the precipitate given by acids with soda-cellulose waste liquor, yielding copper–arsenic derivatives of lignic acid. If necessary, glacial acetic acid is added, in which case the precipitate is separated by decantation after 20 hrs. The product is soluble in water containing a small quantity of sodium carbonate, the solution being very adhesive. For example, a solution of copper sulphate in dilute ammonia is mixed with a suspension of the precipitate produced on acidifying soda-cellulose waste liquor, and ammonium chloride and a solution of arsenious acid in ammonia, and water are added. The precipitate is dissolved in aqueous sodium carbonate solution.

B. FULLMAN.

Treatment of leucite etc. (E.P. 242,336).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Precipitation of calcium sulphite under various conditions, with special reference to sugar factories. R. G. W. FARNELL (J.S.C.I., 1925, 44, 530—532 T, 533—538 T).—Investigation of the precipitation of calcium sulphite from its pure aqueous solutions and solutions of sucrose, diluted cane syrup (15° Brix), and molasses (1%), showed that complete neutralisation of sulphurous acid, long time and high temperature, high initial concentration of sulphurous acid and lime, and absence of colloids were favourable to precipitation. The presence of sucrose had no retarding effect on the precipitation, but greatly diminished the size of the characteristic clusters of crossed needles of $CaSO_3,2H_2O$. The presence of colloids retarded the precipitation and altered the form of the precipitate to spherical granules, the size of which increased with rise of temperature, and with time, p_H, and initial concentration of sulphurous acid. Graphs are given, showing the neutralisation curve H_2SO_3—$Ca(OH)_2$ under different conditions, and the extent of precipitation of $CaSO_3$ at various p_H values. 0·023*N*-calcium hydrogen sulphite has p_H 4·8, and 0·005*N*-calcium sulphite has p_H 9·2. There is little indication of the formation of calcium sulphite and free sulphur dioxide on heating a dilute calcium hydrogen sulphite solution to 100°. The higher a cane juice is sulphured the more complete is the precipitation of calcium sulphite on liming back to a given p_H value. It is suggested that the p_H of the hot juice be increased from 6·8 to 7·6—7·8 in order to obtain more complete precipitation.

Physical examination of sugar juices. K. R. LINDFORS (Ind. Eng. Chem., 1925, 17, 1155—1156; cf. B., 1924, 802).—To obtain indications of the relative efficiency of methods for the treatment of syrups previous to filtration, it is proposed to determine the surface tension (using du Noüy's apparatus), the viscosity (rate of flow from a 100-c.c. pipette), the visible turbidity by means of the Jackson turbidimeter, and the colloidal turbidity by means of Horne's apparatus (*cf.* B., 1924, 694). Such determinations carried out before and after treatment can be of especial value in examining the relative efficacy of various clarification methods and filter-aids.
J. P. OGILVIE.

Temperature and analytical changes in sugar liquor during boneblack filtration. H. I. KNOWLES (Ind. Eng. Chem., 1925, 17, 1151—1152).—When boneblack was filled into a cistern through a funnel set in its mouth, and levelled by hand every hour during the 10—12 hrs. taken to fill, the distribution was irregular throughout, there generally being a much greater proportion of fine material (passing a 50-mesh sieve) in the central portion of the filter. On running liquor at 71° upon char at 66° the temperature in the centre of the filter remained constant for the first 8 hrs., after which it rose rapidly, until after 12 hrs. it had reached about 93°, at which it remained for 8 hrs., then gradually fell. In the outer layers of char the temperature remained more nearly constant during the whole period of 20 hrs., presumably due to the heat absorbed by the walls of the cistern. Liquor in contact with char near the walls was much less decolorised, due probably to the coarser granulation. Decolorisation was comparatively small at the top of the filter; it was greater half-way down, but greatest at the bottom at a point midway between the centre and the walls. It has been found advisable to lime the washed liquor so that it will come off the filter with a p_H of 6·8—7·0, but second liquor may have a p_H as low as 6·5 without apparent loss by inversion.
J. P. OGILVIE.

Purifying molasses by addition of hydrochloric acid. G. DORFMÜLLER and F. TÖDT (Z. Ver. deuts. Zucker-Ind., 1925, 903—913).—The addition of hydrochloric acid to molasses to neutralise the bases present, and obtain a more readily worked product, is not new, having been described by G. F. Meyer in 1876. Experiments are described showing that this addition effects no actual increase in the purity value. If the solution of molasses is dialysed after the hydrochloric acid addition, as in Cutler's method (U.S.P. 1,493,967 and 1,494,218; B., 1924, 609), the economy of the process becomes extremely doubtful.
J. P. OGILVIE.

PATENTS.

Centrifugal apparatus for treatment of sugar liquors and the like. J. J. RAGG (E.P. 233,337, 27.4.25. Conv., 30.4.24).—Syrup or other solution of sugar is introduced into a perforated basket of a centrifugal machine rotating at a high speed within an outer casing, the inner walls of the basket being covered with a layer of filter-cloth, over which is placed a second cloth. Between the two cloths, a sheet of blotting paper, felt, or asbestos, or a thin layer of magnesium carbonate, calcium phosphate, fine coral sand, carbon, kieselguhr, etc., may be interposed.
J. P. OGILVIE.

Separating syrup from residual molasses. C. G. LEONIS (U.S.P. 1,558,554, 27.10.25. Appl., 29.12.24).—Residual molasses is dried and treated with concentrated acetic acid, the sugar thus precipitated being separated and washed with alcohol.
J. P. OGILVIE.

Vacuum pan [for graining sugar]. C. WHITE (U.S.P. 1,558,957, 27.10.25. Appl., 18.3.25).—A vacuum pan for graining sugar is provided with the floor sloping downwards towards the centre, the heating element within the casing being adjacent, but spaced from, the floor. A central open space extends upwards through the heating element, this constituting a well having an upper and a lower opening with a closed side-wall. A core projects upwards from the casing floor through the well, forming a displacement member whereby the volume of liquid within the well is reduced.
J. P. OGILVIE.

Dextrose hydrate. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P.

1,559,176, 27.10.25. Appl., 16.4.24). — Normal dextrose hydrate is heated so that some of the water of crystallisation is driven off, without impairing the form of the crystals. When exposed to a damp atmosphere partially dehydrated crystals absorb water until fully hydrated without becoming moist, and on this account are suitable for storage.
T. S. Wheeler.

Recovering diatomaceous earth. (U.S.P. 1,561,042).—See VIII.

XVIII.—FERMENTATION INDUSTRIES.

Steam-boiling of brewery worts. E. Willemart (Chim. et Ind., 1925, **14**, 686—690).—Unsatisfactory results with steam are frequently due to technical faults in the plant. To obtain maximum utilisation of the heat, a tubular heater should be used with short tubes of small diameter arranged as nearly horizontal as practicable. Open coils are more satisfactory than closed coils, the latter having a much lower coefficient of heat-transmission, and in addition the circulation of steam is imperfect. The steam-jacketed copper is the least satisfactory; the wort is not properly circulated and the " break " is incomplete, though cold storage of low-fermentation beers helps to correct this. Agitation allows of a more vigorous boil without risk of the contents boiling over; 10% more evaporation can be obtained, and the deposit from an agitated wort is greater than from one boiled without agitation, the wort is clearer, and the resulting beer brighter and more resistant to infection. The use of high pressures of steam (70—130 lb./in.^{2}) is beneficial. Boiling under pressure is attended with good results, but if the pressure exceeds 21 lb./in.^{2}, the flavour of the beer is impaired owing to the denaturation of the bouquet substances in the hop. This trouble can sometimes be prevented by addition of sulphuric acid to the wort. The acidity of wort is frequently insufficient to promote the optimum " break " upon boiling. The addition to wort of 0·03% of phosphoric or 0·005% of sulphuric acid more than doubled the bulk of the precipitate upon boiling and the clarity of the boiled wort was improved; sulphuric acid was slightly superior to phosphoric acid in both respects. An economy of steam and time may be effected by commencing to heat the wort vigorously as soon as the first portion is run into the copper, as with modern mashing plant it is unnecessary to allow diastatic action to take place in the copper when the weak worts are run in. G. T. Peard.

Patents.

Producing lactic acid and/or nitrogen-containing lactic acid compounds from sugar-containing raw materials by means of micro-organisms. A. Pollak (E.P. 222,514, 29.9.24. Conv., 29.9.23).—In the production of lactic acid for utilisation in the manufacture of yeast, wort made from sugar-containing raw materials is mixed with a relatively large amount of a lactic acid bacteria "starter" and run through a series of vats, to one or more of which basic nitrogenous substances are added to neutralise the acid and form the source of nitrogen for the yeast. The process is continuous. The acidified wort may be sterilised by boiling, or it may be directly pitched with yeast, the acid being neutralised with basic substances as it is formed.
G. T. Peard.

Stock food (U.S.P. 1,561,158).—See XIXa.

XIX.—FOODS.

Chemistry of bread. New Zealand wheat flours and fermentation. W. L. M. Dearsley (N.Z. J. Sci. Tech., 1925, **8**, 34—38).—Chemical and baking analyses of fine typical strains of New Zealand wheat, which are quoted, resulted in the following conclusions:—The order of acidities (calculated as acetic acid) of the flours is that of their p_H values; the hydration of the gluten is greater the nearer the p_H is to 5·0. The water-absorption value of the flours depends upon both quantity and quality (*i.e.*, hydration) of gluten, and one flour may be more satisfactory than another containing a greater total amount of gluten. A dough of lower initial p_H ferments faster than one of higher p_H, and the bread will have a better texture and colour, though the latter is to some extent dependent upon the fat present; in all cases the p_H approaches 5·0 after fermentation. The volume of the loaf is connected with the water absorption capacity of the flour; the losses of weight in baking and upon keeping, due to loss of water, are less the greater the hydration of the gluten. The question of "strong" and "weak" flours may perhaps be explained by a consideration of hydrogen-ion concentrations. It is probable that the best flour is one which has a high content of gluten and which will yield a dough having p_H as near 5 as possible. Bread having p_H greater than 5·1 is liable to attack by *B. mesentericus* (a "ropy" organism). G. T. Peard.

Influence of feeding coconut oil-cake [to cows] on the Polenske value [of the butter]. Paraschtschuk (Milchwirts. Zentr., 1925, **54**, 161—163).—Feeding experiments with individual cows show that the increase in the Polenske value of the butter which follows addition of coconut oil-cake to the ration is much less when the cows are in poor condition than when they are in normal condition. C. T. Gimingham.

Cacao beans and cacao products. III. H. Fincke (Z. Unters. Nahr. Genussm., 1925, **50**, 205—220; cf. B., 1924, 489; 1925, 50).—Cacao beans, smaller than the average size in a number of samples, were found to have a lower percentage of fat and larger amount of husk and germ. The fat content of cocoa pastes, calculated on a dry-matter basis, was slightly increased by roasting the beans. The fat content of the husks was 2—3% and differed from the fat of the kernels in having a higher iodine value, refractive index, and acidity. The last-named was particularly pronounced. The

fat of the cacao germ was similar in its properties, but had an unusually low saponification value and a setting point which was ill-defined. It was unpleasant in taste and smell and of a dark brownish colour. The presence of fat from the husk and germ in cacao butter is best indicated by the increased acidity and, to some extent, by the taste and smell. Adulteration of cocoa powders by husk and germ can be detected to some extent by examination of the residue after sedimentation, and comparative figures are given. Increased fibre content and acidity of the fat are the most notable points.
A. G. POLLARD.

Acid- and base-forming elements in foods. G. W. CLARK (J. Biol. Chem., 1925, **65**, 597—600).—Figures are given for the sodium, potassium, calcium, magnesium, chlorine, phosphorus, sulphur, and nitrogen content of the ash of numerous foods.
C. R. HARINGTON.

Is antirachitic factor [vitamin-D] of cod liver oil when mixed with grains, destroyed through storage? E. B. HART, H. STEENBOCK, and S. LEPKOVSKY (J. Biol. Chem., 1925, **65**, 571—578).—Cod liver oil when mixed in the proportion of 0·5% with maize meal retained its antirachitic properties during storage at the ordinary temperature in unsealed vessels for six months. C. R. HARINGTON.

Development and composition of potato plants. HUXDORFF.—See XVI.

PATENTS.

Deodorising, cooling, and dehydrating fluid substances [milk etc.]. TE AROKA DAIRY CO., LTD. (E.P. 220,627, 11.8.24. Conv., 14.8.23).—The warm fluid is led from a pasteuriser or preheater into a vacuum vessel and the ebullition set up causes concentration, deodorisation, and cooling. The odour in milk and cream due to a diet of turnip and rape etc. is completely removed, and the acidity of milk is decreased. By making the process continuous, condensed milk can be produced. The spherical vacuum vessel is fitted with a central water condenser, having the shape of an inverted frustum of a cone, also with large blades or sweeps shaped to the container walls and baffle plates near the top, but below the mouth of the condenser. The plant is sterilised and the vacuum obtained by blowing in steam and condensing it. The fluid is given a circulatory and upward movement by the sweeps and then deflected downwards by the baffle plates over the outer wall of the condenser. Charging and discharging are carried out automatically without releasing the vacuum. G. T. PEARD.

Baking powder. R. E. JONES (U.S.P. 1,558,888, 27.10.25. Appl., 14.2.25).—A baking powder comprising acid calcium phosphate, sodium bicarbonate, maize starch, and taka-diastase is claimed. When mixed with flour the diastase transforms a portion of the starch into sugar, giving the bread a sweet taste. T. S. WHEELER.

Process of and composition for dough-making. B. LOWY (U.S.P. 1,559,330, 27.10.25. Appl., 18.5.25).—Condensed buttermilk (100 pts.) with a high content of lactic acid is mixed with calcium phosphate (1·25 pts.) at 40° to produce a calcium lactophosphate, ammonium tartrate (0·75 pt.) is added, and the mixture is dissolved with yeast, salt, and sugar in water. 100 pts. of flour are added for each part of the prepared mixture and the whole is formed into dough. During fermentation the carbon dioxide evolved in presence of ammonium tartrate, which acts as a catalyst, liberates lactic acid from the lactophosphate. This mellows the gluten cells and permits the entrance of carbon dioxide, so improving the quality of the loaf. T. S. WHEELER.

Semi-solid stock food. W. P. M. GRELCK (U.S.P. 1,561,158, 11.11.25. Appl., 27.9.23).—The starch in a ground cereal is gelatinised by heating with water and the product mashed with malt. The wort is fermented to lactic acid and mixed with grain to form the food, in which the lactic acid acts as a preservative. G. T. PEARD.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Influence of manures on the yield and morphine content of the latex of the opium poppy. H. E. ANNETT and H. D. SINGH (Mem. Dept. Agric. India, Chem. Series, 1925, **8**, 27—37).—Organic manures such as oil-seed cakes and cattle manure increase the yield of opium and its morphine content, the increase being due to the phosphoric acid in the manure and not to the nitrogen or potash. Superphosphate caused an increase in the morphine content of the opium. C. O. HARVEY.

Ash constituents of Indian opium. H. E. ANNETT and M. N. BOSE (Mem. Dept. Agric. India, Chem. Series, 1925, **8**, 45—51).—The amount of ash constituents of the latex of the opium poppy rises at each successive lancing up to the fourth, whereas there is a rapid fall in morphine content and, to a lesser extent, a fall in narcotine content. The codeine and papaverine contents remain more or less constant, as also does the composition of the ash. Little or no increase in ash occurs in later lancings, and its composition is not appreciably affected by manuring. Sulphate and phosphate in the original opium rapidly increase, the former up to the third and the latter up to the fifth successive lancing. Chlorides were not found in the latex.
C. O. HARVEY.

Electrometric titration of alkaloids and the reaction of alkaloid salts. I. M. KOLTHOFF (Pharm. Weekblad, 1925, **62**, 1287—1293).—Recent methods are discussed, and a table of dissociation constants and p_H values for a great number of alkaloids and salts is given, from which suitable indicators can be chosen for particular titrations, where colourless aqueous solutions are available. Only when the solutions are coloured or a suitable

indicator is not available do electrometric methods offer any advantage. S. I. LEVY.

Sterilisation of tropacocaine hydrochloride and its solution. S. MATSUNAMI (J. Pharm. Soc. Japan, 1925, [520], 6—7; Chem. Zentr., 1925, **96**, II., 2069).—Heating for 1 hr. at 130° does not decompose tropacocaine hydrochloride; heating for 2 hrs. at 135—140° decomposes it very slightly. A 2% solution sterilised as usual yields (per 10 g. of hydrochloride) 0·0004—0·0005 g. of benzoic acid and 0·0006—0·0007 g. of ψ-tropine. The aqueous solution of the latter decomposes only on heating at 170—180° (tube), yielding a trace of tropidine. The alkali of the glass promotes the hydrolysis of tropacocaine hydrochloride solution. The hydrochloride and its solution are stable to sunlight and ultra-violet light. B. FULLMAN.

Comparative experiments on the narcotic and toxic effects of some halogen derivatives of hydrocarbons. J. MÜLLER (Arch. exp. Path. Pharm., 1925, **109**, 276—293).—White mice were subjected to the action of known concentrations of various halogen derivatives, chloroform in a concentration of 0·027 g./litre being used as a standard. The order of increasing narcotic activity of the substances examined was: propyl chloride, ethyl bromide, methylene chloride, ethylidene chloride, propyl bromide, dichloroethylene, carbon tetrachloride, ethylene chloride, chloroform, and tetrachloroethane. The order of increasing toxicity was: propyl chloride, dichloroethylene, ethylidene chloride, methylene chloride, propyl bromide, ethylene chloride, ethyl bromide, chloroform, carbon tetrachloride, and tetrachloroethane. Histological examinations were made in each case. S. I. LEVY.

Identification of adaline. C. GENOT (Chim. et Ind., 1925, **14**, 679—685).—Adaline (bromodiethylacetylcarbamide), a white crystalline powder, m.p. 117—118°, is frequently adulterated with cheaper substances (veronal etc.). It is almost insoluble in water (0·05%) and in formic, perchloric, and hydrobromic acids. Its colour reactions and microcrystalline reactions and the refractive indices of its solutions in the usual solvents, are given in great detail, together with a table of melting points of mixtures of adaline with adulterants in various proportions. The microcrystalline forms are characteristic and divisible into four classes:—fine needles are deposited from water, ethyl and amyl alcohols, chloroform, ether, ethyl bromide, fuming nitric acid, and concentrated hydrobromic acid; short needles grouped like an ear of barley from dilute hydrochloric acid, xylene, and butyric acid; large prisms from benzene, acetyl chloride, acetic anhydride, methyl alcohol, light petroleum, aniline, and nitrobenzene; very short and fine needles, arborescent or in star-like clusters, from dilute ammonia, carbon disulphide, acetone, toluene, and acetic acid. Photomicrographs of many of these forms are reproduced. G. T. PEARD.

Determination of small quantities of halogens [chlorine in benzaldehyde] and sulphur [in mineral oils]. O. WAGNER (Z. angew. Chem., 1925, **38**, 1068).—Chlorine in the proportion of about 0·2% in, *e.g.*, benzaldehyde may be accurately determined by treating the material with pure fuming sulphuric acid in a Carius bomb tube, and then determining the chloride as silver chloride by the micro-method of Pregl. This yields results accurate to within 0·02%. Sulphur is determined by treatment with pure fuming sulphuric acid and precipitation with barium chloride, the barium sulphate being collected in a quartz filter-tube with attached siphon and ignited in a micro-bomb furnace (*ibid.*, 1923, **36**, 494). B. W. CLARKE.

Testing *Spt. Aether Nit.* J. RAE (Pharm. J., 1925, **115**, 633—634).—The nitrite content is determined colorimetrically by means of a dilute hydrochloric acid solution of sulphanilic acid, the coloration being developed by the final addition of a dilute solution of ammonia, and compared with a permanent potassium dichromate standard. C. O. HARVEY.

Studies on camphor group. I. Formation of camphor from turpentine oil. Y. MURAYAMA (J. Pharm. Soc. Japan, 1925, [524], 882—884; *cf.* B., 1924, 731).—Bornyl oxalate prepared by heating a mixture of anhydrous oxalic acid, turpentine oil, and ferric chloride in carbon tetrachloride solution gives either *d*- or *l*-borneol on saponifying with alcoholic potash according to the nature of the turpentine oil used. On oxidation with a mixture of sulphuric and nitric acids, *d*- or *l*-borneol gives *d*- or *l*-camphor, $[\alpha]_D^{17} + 15{\cdot}00°$ or $[\alpha]_D^{18} - 11{\cdot}30°$ respectively. Camphor is also formed from camphane-2-carboxylic acid by oxidation with permanganate in alkaline solution. The product melts at 175° and its semicarbazone at 235°. K. KASHIMA.

Determination of menthol in alcoholic solutions. DONATH and SEBOR (Chem. Listy, 1925, **19**, 363—364).—The percentage content of menthol in alcohol is given by the formula $p=0{\cdot}35a$ (where a is the polarimeter reading for 100 c.c. in a 200-mm. tube). The strength of the alcohol is determined by its specific gravity, making a correction of +0·0004 for every gram of menthol present. B. W. ANDERSON.

Action of phosphoric acid on certain terpenes and related compounds. P. G. CARTER, H. G. SMITH, and J. READ (J.S.C.I., 1925, **44**, 543—547 T). —By the action of syrupy phosphoric acid (*d* 1·75) diterpenes or diterpene mixtures have been obtained from *l*-α-phellandrene, *d*- and *l*-pinene, *d*-limonene, cineole, and the phellandrene fractions of the essential oils of *Eucalyptus dives*, *E. phellandra*, and *Melaleuca acuminata*. With the exception of the diterpene from *l*-α-phellandrene which has $[\alpha]_D^{20}$ up to $+ 21{\cdot}20°$, and the eucalyptus oil fractions, the diterpenes are almost optically inactive. It is suggested that the polymerisation occurs by hydration of the terpene, followed by dehydration from one or two molecules, giving in the first case α- and γ-terpinene, and in the second, the diterpene; terpin

hydrate was isolated from the product of the incomplete reaction between cineole and phosphoric acid. The diterpenes are viscid, pale yellow oils, b.p. in the neighbourhood of 192—194°/24 mm., n_D^{20} 1·5157—1·5186, d_4^{20} 0·9236—0·9315; they are insoluble or only slightly soluble in hydroxylic solvents, but are readily miscible with chloroform, ether, ligroin, etc. No solid derivatives could be obtained by the action of bromine, hydrogen chloride, nitrous acid, nitrosyl chloride, and other reagents applied to olefinic compounds, with the exception of ill-defined, yellow nitro-products. The variation with temperature of the density and refractive index of the diterpene are given, and the physical constants of the diterpenes from the various sources are tabulated.

Examination of some Rhodesian eucalyptus oils. P. G. CARTER and J. READ (J.S.C.I., 1925, **44**, 525—526 T).—The physical constants of the oils of *E. Macarthuri*, *E. citriodora*, and *E. globulus* from seedlings two to three years old, grown in Northern Rhodesia, show appreciable differences from those of Australian oils of the corresponding species. The amounts of the chief components of commercial value in the oils from the two sources are almost identical, with the exception of the geranyl acetate content of Rhodesian oil of *E. Macarthuri*, which is markedly low; the same oil contains a larger quantity of eudesmol than is usual. The yields of oil are much higher for the Rhodesian than for the Australian material.

Blue oil of lignite tar. HERZENBERG and RUHEMANN.—See III.

PATENTS.

Making crotonaldehyde. E. C. R. MARKS. From CARBIDE AND CARBON CHEMICALS CORP. (E.P. 242,521, 31.3.25).—Aldol prepared and stored in an atmosphere of nitrogen, acetylene, or other non-reacting gas, is heated, preferably under reduced pressure, to a temperature not much above 165°, and crotonaldehyde is removed as vapour as fast as it is formed, by passing a current of steam or a non-reacting gas through the liquid. The crotonaldehyde is condensed and stored with the exclusion of air, and acetaldehyde mixed with it is removed by fractional distillation in the presence of a non-reacting gas. The process may be worked continuously by passing a slow stream of aldol into a reaction vessel maintained at a suitable temperature.

L. A. COLES.

New acid and its salts strongly absorbing ultra-violet rays. T. SUZUKI and S. SAKURAI (E.P. 242,721, 18.8.24).—The sodium or potassium salt is prepared by condensing dextrose or inverted sugar with phenylhydrazine-*p*-sulphonic acid in presence of sodium or potassium acetate at 100°. The free acid is prepared by precipitation of the lead salt from aqueous solution and passing hydrogen sulphide through a suspension of the precipitate. The acid and its neutral, especially alkali, salts are readily soluble in water. A 2 cm. layer of 0·01% solution completely absorbs the ultra-violet, and passes most of the visible rays, absorbing, however, some of the rays from the G and H lines to the ultra-violet. Used in a gelatin filter, 2 g. per sq. m. completely absorb the ultra-violet. Compared with Filter Yellow K for equal visual absorption, the new substance is a more efficient absorber of the ultra-violet. Used in conjunction with Tartrazine in the amount of 2 g. of Tartrazine and 1 g. of the salt of the new acid per sq. m., filters far more efficient than those made with Tartrazine alone can be prepared. A red filter of Rose Bengale and Tartrazine cuts off all ultra-violet if some of the new salt is added, and improved blue filters are prepared using the new salt with Toluylene Blue etc. The insoluble basic lead salt of the acid provides an ultra-violet ray-proof paint, and fibres can be treated with the acid if lead is used as mordant.

W. CLARK.

Manufacture of benzoic acid from benzotrichloride. Manufacture of benzoyl chloride. A. GEORGE, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,557,153–4, 13.10.25. Appl., 22.7.24).—(A) Benzotrichloride containing zinc chloride in suspension is heated with agitation at 100—120°, and water is added at the rate at which it reacts. Benzoic acid is rapidly formed in almost theoretical yield. (B) By suitably reducing the quantity of water added in the above reaction the hydrolysis may be stopped when substantially pure benzoyl chloride is present. It is removed by distillation under reduced pressure and is obtained in 85% yield.

T. S. WHEELER.

Manufacture of diphenylaminechloroarsine. W. L. TANNER, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,557,384, 13.10.25. Appl., 17.8.22).—An equimolecular mixture of diphenylamine and arsenic trichloride heated to 210° gives diphenylaminechloroarsine in theoretical yield. T. S. WHEELER.

Manufacture of urea [carbamide] from ammonium carbamate. N. W. KRASE and V. L. GADDY, Assrs. to R. C. TOLMAN (U.S.P. 1,558,185, 20.10.25. Appl., 25.3.21).—In the production of carbamide by heating a mixture of ammonium carbonate and ammonium carbamate in an autoclave, the mixture is first briquetted, to facilitate charging and discharging of the autoclave.

T. S. WHEELER.

Irritant product from the *Rhus* plant. P. MASUCCI, Assr. to H. K. MULFORD CO. (U.S.P. 1,559,340, 27.10.25. Appl., 16.4.23).—The leaves of *Rhus toxicodendron* are extracted with benzine, and the extract is decolorised with fuller's earth and evaporated to dryness. The product is a wax containing the irritant principle of the plant, and is of therapeutic value. T. S. WHEELER.

Making lead tetra-alkyl. W. S. CALCOTT, Assr. to E. I DU PONT DE NEMOURS AND CO. (U.S.P. 1,559,405, 27.10.25. Appl., 5.10.22).—A finely-divided alloy of lead (131 pts.) and sodium (29 pts.) is mixed with ethyl bromide (135 pts.) and triethylamine (19·2 pts.) or other catalyst for the Grignard

reaction, and water (4 pts.) is dropped in slowly during 16 hrs. at a temperature below 35°. A large excess of water is then added, and lead tetraethyl separated by distilling in steam. T. S. WHEELER.

Oxidising organic compounds [*e.g.*, **toluene to benzaldehyde**]. J. V. MEIGS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,560,297, 3.11.25. Appl., 4.4.19).—Ferric chromate and ammonium vanadate are mixed and heated to give an iron vanadochromate in granular form which is saturated with silver nitrate solution, dried, and treated with hydrogen so that metallic silver is formed. The product is placed in a chamber at 250° and toluene vapour mixed with steam and air is passed over it. The temperature and quantity of steam are regulated so that the quantity of carbon dioxide in the exit gases remains at about 3%. A good yield of benzaldehyde is obtained.
T. S. WHEELER.

Preparation of bismuth oleate. F. HOFFMANN-LA ROCHE U. Co. (Swiss P. 106,664, 18.8.23).—Hydrated bismuth oleate is prepared as a soft soap by adding, at 15°, bismuth nitrate dissolved in 13% mannitol solution to oleic acid in 80% alcohol neutralised with the calculated quantity of aqueous sodium hydroxide (alkali oleates in general are also specified). The precipitate is washed free from nitric acid. The anhydrous oleate is obtained by evaporation of an ether solution of the oleate in a vacuum. It is insoluble in water but easily soluble in organic solvents. For therapeutical purposes it is used as an oily emulsion, prepared by mixing oil with the ether solution of the hydrated form, and distilling off the ether and water. B. FULLMAN.

Making alkyl chlorides. W. R. WEBB, Assr. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,560,625, 10.11.25. Appl., 18.7.24).—Hydrogen chloride rapidly reacts with an olefine when passed with it over aluminium chloride at about 150°, to give the corresponding alkyl chloride in satisfactory yield. T. S. WHEELER.

Production of cyanamide solutions. S. J. GELHAAR (Swed. P. 57,109, 23.12.21, 57,110, 16.2.22, and 57,111, 5.4.22).—Calcium cyanamide is finely ground with water or with a solution of cyanamide or carbamide, with, if necessary, sufficient sulphuric acid, carbon dioxide, or the like to neutralise the lime present. The calcium cyanamide may be mixed with acid and such a quantity of water that a solid product is obtained, and this extracted with water or a solution of cyanamide or carbamide. Alternatively the calcium cyanamide is treated with water at a temperature not above 125° so that water not present in the form of hydrate is vaporised. Thereby undesirable sulphur and phosphorus compounds are decomposed, calcium oxide is converted into the hydroxide, and calcium cyanamide is obtained in the form of a very fine powder, which is extracted in presence of carbon dioxide or gases containing carbon dioxide. J. S. G. THOMAS.

Organic acids from soda-cellulose waste liquor (F.P. 563,747).—See V.

Stabiliser for solutions containing loosely-combined oxygen (U.S.P. 1,559,600).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Treatment of felted materials, papers, and the like [for photographic purposes]. KODAK LTD., and W. G. BENT (E.P. 241,769, 20.1.25).—Rubber latex is added to the coating mixture in the manufacture of coated papers and the like. A suitable mixture is made as follows: 400 lb. of blanc fixe or barytes in paste form (containing 25% of water) are mixed with 10 gal. of water and added to 50 lb. of gelatin dissolved in 30 gal. of water; 1500 c.c. of saturated alum solution are then run slowly in, followed by rubber latex (containing 35—40% of non-volatile constituent) in the proportion of 0·5 gal. of latex per 640 lb. of mixture. Colouring agents may be added if desired. Paper coated by this process is particularly suitable for photographic purposes, since the final sensitised paper is resistant to water and shows no tendency to crack. D. J. NORMAN.

Natural colour kinematography. K. CAMPBELL (E.P. 242,727, 20.8.24).—Panchromatic film is coated on the emulsion surface with a multi-colour screen consisting of fine pollen or spores, the latter preferably of *L. clavatum*, stained in the three primary colours. The screen is prepared by bleaching the pollen or spores with hydrogen peroxide solution acidified with hydrochloric acid at 57—63°, drying at the same temperature, pulverising, and staining equal weights of the bleached substance with the alcoholic dye solutions necessary to give mono-spectrum yellow, mono-spectrum red, and mono-spectrum blue. The stained and dried powders are mixed in equal proportions in gelatin or celluloid and coated to give a closely-packed single layer of coloured grains on the emulsion surface of the film. Exposure is through the screen layer, and the exposed film may be developed to a negative and printed on a positive film carrying a similar multi-colour screen, or else be reversed to give a direct positive. In printing, the colour-screen layer of the positive is maintained in close contact with the back of the negative film. The multi-colour screen may similarly be applied to panchromatic plates. W. CLARK.

Solution for sensitising paper, glass, fabric, etc. M. MICHELS (Swiss P. 109,722, 22.4.24).—A solution containing ferrous ammonium citrate, silver nitrate, and tartaric acid is heated at 50—60° for 6—8 hrs. and then separated from any small precipitate which forms. For special printing processes, copper sulphate, uranyl nitrate, or chloral hydrate may be added. W. CLARK.

Acid and salts strongly absorbing ultraviolet rays (E.P. 242,721).—See XX.

XXII.—EXPLOSIVES; MATCHES.

Stability of nitrocelluloses. Duclaux.—See V.

Patents.

Explosive compound [trinitrophenoxyethyl nitrate]. H. A. Lewis, Assr. to E. I. du Pont de Nemours and Co. (U.S.P. 1,560,426, 3.11.25. Appl., 26.1.25).—2:4-Dinitrochlorobenzene (100 pts.) is dissolved in ethylene glycol (322 pts.) and an equal quantity of the glycol mixed with caustic soda (20 pts.) and water (12 pts.) is added. On heating the mixture on the water bath, dinitrophenoxyethyl alcohol is formed and separates on pouring into water. One part is dissolved in 10 pts. of 95% sulphuric acid and slowly added to 3 pts. of concentrated nitric acid at 30° and heated to 50° for 1 hr. On pouring into water *trinitrophenoxyehayl nitrate*, m.p. 105°, is obtained. It is a valuable high-explosive. T. S. Wheeler.

Explosive [trinitrophenylnitroaminoethyl nitrate]. R. C. Moran, Assr. to E. I. du Pont de Nemours and Co. (U.S.P. 1,560,427, 3.11.25. Appl., 26.1.25).—100 pts. of 2:4-dinitrochlorobenzene are dissolved in 510 pts. of 95% alcohol, 30 pts. of aminoethyl alcohol are added, and the mixture is heated to 70° and treated with 20 pts. of caustic soda in 30 pts. of water. On concentration dinitrophenylaminoethyl alcohol separates. When dissolved in 10 pts. of 95% sulphuric acid and added to 3 pts. of concentrated nitric acid at 30° and heated at 50° for 1 hr. *trinitrophenylnitroaminoethyl nitrate*, m.p. 125°, is obtained. It is a valuable high explosive. T. S. Wheeler.

XXIII.—SANITATION; WATER PURIFICATION.

Sterilisation of water by chlorine and some of its compounds. C. H. H. Harold (J. Roy. Army Med. Corps, 1925, 45, 190—207; Pub. Health Eng. Abstracts, Sept. 19. 1925. W 54; cf. B., 1924, 886).—Prior addition of 0·25—0·5 p.p.m. of ammonia increased the germicidal action of chlorine, reduced loss of chlorine by absorption, and produced a water with satisfactory taste. Good results were obtained only when the ratio of chlorine to ammonia was 4:1. The germicidal power of hypochlorous acid is lower than that of chlorine, and the former is more readily absorbed by water. Chloramine was found to possess the highest germicidal power of any chlorine-ammonia compound, and the rate of sterilisation though slower than with chlorine, was not reduced by a moderate content of colloidal matter. R. E. Thompson.

Endo's medium [for *B. coli*]. N. MacL. Harris (Canad. Pub. Health J., 1925, **16**, 326).—In experiments made with a view to improve the stability of Endo's medium the best results were obtained with "Difco" bacteriological peptone and with a basic fuchsin consisting of approximately equal proportions of rosaniline and pararosaniline. Substitution of dipotassium phosphate (0·3—0·5%) for meat extract in the formula of the standard method of the Amer. Pub. Health Assoc. was found advisable. R. E. Thompson.

Importance of differentiating colon-aerogenes group in examining water. J. J. Hinman, jun. (Amer. J. Pub. Health, 1925, 15, 614—619).—A discussion of the test for *B. coli* in water, with special reference to methods for differentiation of the colon-aerogenes group and the value of information derived therefrom. In the examinations of treated water from Iowa plants during the period 1914 to 1924 inclusive, of 18,847 culture tubes 31·5% gave positive presumptive tests, 9·4% of these being due to presence of *B. coli*, 9·8% to *B. aerogenes*, and 80·8% to other gas formers. R. E. Thompson.

Utilisation of sewage for gas production. Sierp (Gas u. Wasserfach, 1925, **68**, 772—776).—A fuel gas can be produced from sewage sludge by suitably controlled bacterial action. Some aspects of the operation of the process at Essen are described. The crude gas contains 65—95% CH_4, 5—35% CO_2, 0—8% H_2, 0—5% N_2, and 0—0·25% H_2S. After purification the gas contains 90—98% CH_4. The hydrogen is relatively high during the initial period of fermentation, but later, under the influence of particular bacteria, it reacts with carbon dioxide to form methane. Too great a dilution of the sludge gives rise to acid fermentation and a higher percentage of hydrogen sulphide. The optimum temperature is about 25°, and in cold weather an increased yield may be obtained by heating the fermentation tanks. The sewage must be free from bacterial poisons, such as may occur in the waste from gas works, copper works, tanneries, etc. The collection of the gas gives rise to certain mechanical difficulties. These have been overcome by the use of deep fermentation tanks, the gas outlet of which is fitted with a device for allowing the passage of gas while preventing that of the scum. The gas collects in a hood covering the outlet, whence it is withdrawn as required. The gas is used as a constituent of town gas, as a fuel gas on the works, or is compressed into cylinders. Various Essen works produce gas in this way in amounts ranging from 250,000 to 600,000 m.³ per annum. A. B. Manning.

Pitting in iron water pipes. Liebreich.—See X.

Corrosion of water pipes. Cassel.—See X.

Natural water corrosion. Baylis.—See X.

Tuba (*Derris*). Gater.—See XVI.

Patents.

Water distilling apparatus utilising exhaust gases. C. Day (E.P. 242,328, 25.3.25).—In a distilling apparatus utilising exhaust gases from internal-combustion engines, furnaces, etc., a constant level of water is automatically maintained in the evaporating vessel, and the amount of cooling water is regulated by means of a thermostat device. C. O. Harvey.

Sulphur fungicide (U.S.P. 1,559,984).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEBRUARY 5, 1926.

I.—GENERAL; PLANT; MACHINERY.

Measurement of temperature by thermocouples in unequally heated enclosures. W. MANDELL (Proc. Physical Soc., 1925, 38, 47—68).—A consistent difference in the temperature recorded by thermocouples of different thickness has previously been observed. Further experiments with four couples of different diameter in a gas enclosed between two plates at different temperatures confirm the observation. The thickest couple recorded the highest temperature near the hot plate, and the thinnest the highest temperature near the cold plate. The recorded temperature varies with different gases, being nearest the true value in hydrogen. The temperature of a couple of zero diameter, found by extrapolation, was taken as the true temperature. When the temperatures of the plates differed by 200° in air the difference between the temperature of the zero couple and of the thickest was 20°. The walls of the vessel also affect the recorded temperature. All the couples agreed at some temperature between the plates, this being nearer the hot plate with black, than with bright, surfaces. The results are explained by radiation and molecular impact effects. The conclusion is reached that thermocouples cannot be used as precision instruments in industrial plant except under black-body conditions of radiation. C. J. SMITHELLS.

Application of spectro-photography to measurement of high temperatures. I. O. GRIFFITH (Proc. Physical Soc., 1925, 38, 85—87).—The spectrum of the source is photographed through a neutral wedge placed immediately in front of a photographic plate. The curved boundary of the spectrum varies in height, the variation depending upon the distribution of energy in the source, and therefore upon its temperature. It is assumed that the source radiates as a black or grey body, but with certain limitations the method is applicable to bodies which are neither black nor grey. The probable error is 40° at 5000° and 150° at 10,000°. C. J. SMITHELLS.

Photo-electric radiation pyrometer. F. A. LINDEMANN and T. C. KEELEY (Proc. Physical Soc., 1925, 38, 69—73).—By means of two photo-electric cells with suitable colour filters the energy radiated in two distinct spectral bands is compared. By combining the ascertained sensitivities of the cells and their filters with the radiation law the black-body temperature of the radiator can be determined. The accuracy of measurement is 1·6° at 2000°, 3° at 3000°, and 7° at 4000°. C. J. SMITHELLS.

Gallium-in-quartz thermometer graduated to 1000°. S. BOYER (Ind. Eng. Chem., 1925, **17**, 1252—1253).—The b.p. of gallium is 1700° and when highly purified and sealed in quartz tubes under 2 or 3 μ pressure the metal may often be cooled to −15° or −20° before solidifying. In order to prevent wetting of the quartz thermometer capillary tube, oxide films, occluded gas (present in electrolytically prepared gallium), and any traces of zinc or arsenic must be absent. Oxide is removed by hydrochloric acid, the bright metal is solidified under acid, the excess of acid removed, and the gallium transferred to the vacuum apparatus where the protective surface film of chlorides is removed by volatilisation, and the metal introduced into the attached thermometer tube. In order to etch the thermometer stem polymerised tung oil is used as a protective coating. The etched marks are coloured greyish-black by a mixture of copper oxide and sand. D. G. HEWER.

Measurement of humidity in closed spaces. E. GRIFFITHS (Engineering Committee of the Food Investigation Board, Special Report No. 8, 1925, 54 pp.).—An account is given of experiments made with existing types of hygrometers, together with a description of novel instruments and methods of hygrometry designed to meet special requirements. Various methods of controlling the humidity in closed spaces are also described. J. S. G. THOMAS.

See also A., 1926, 12, **Examination of nickel catalysts with X-rays** (CLARK, ASBURY, and WICK). 19, **Adsorption by coconut charcoal from alcohol–benzene and acetone–benzene mixtures** (TRYHORN and WYATT). 41, **Automatic pressure regulator** (DAWSON); **Viscosity measurements by means of Mohr's balance** (STICH); **Circulation apparatus for gases** (CHATTERJI and FINCH).

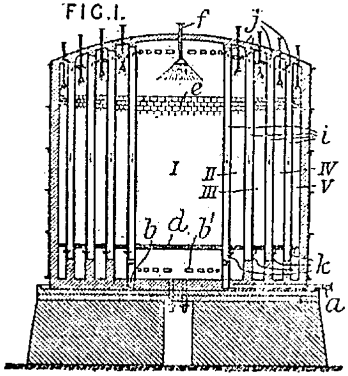
Gas masks. ENGELHARD.—See XXIII.

PATENTS.

Absorption apparatus for gases and vapours. CHEM. WERKE LOTHRINGEN G.M.B.H. (E.P. 236,918, 24.6.25. Conv., 10.7.24).*—Gases or vapours are absorbed by a liquid in a series of towers, I to V, one within the other. The gases are introduced through the pipe, *a*, and circular channel, *b*, having apertures, *b′*, and pass upwards through the perforated plate, *d*, and filling material, *e*, in the tower, I, absorbent liquid being sprayed in through *f*. The gases pass from each tower to the next outer one through annular passages, *i*, and apertures, *j*, *k*.

The apparatus is specially suitable for absorbing nitrous gases, in which case the outermost tower is supplied with an alkaline solution to absorb the last traces of nitrous gases. S. Binning.

FIG. 1.



Colloid mills, and drying, concentrating, mixing, emulsifying, grinding, and like machines. Rissik, Fraser and Co., Ltd., and A. Fraser (E.P. 243,052, 16.8.24).—A colloid mill consisting of a pair of discs, preferably with concentric, intercalating corrugations, rotating at high speed in opposite directions, is provided with a main feed of material at the centre of the discs, also with an auxiliary feed to a zone between the centre and circumference, the passages for this being formed in the thickness of one disc. The portion of the discs between the centre and the auxiliary inlets may be provided with teeth (interrupted corrugations) to provide a preliminary grinding action upon the material that enters by the main feed. Instead of two discs rotating in opposite directions, there may be one fixed and one rotating disc. B. M. Venables.

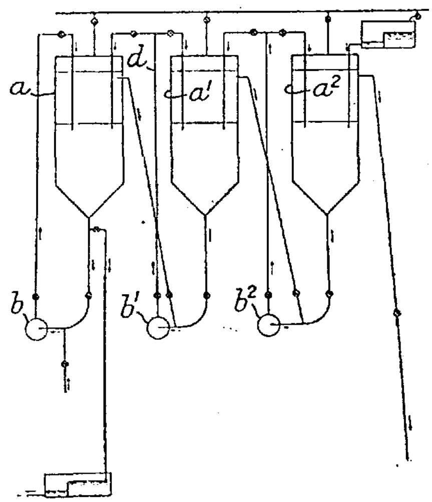
Grinding, milling, and pulverising apparatus. R. K. Ramsay and M. J. Mayhew (E.P. 243,166, 28.11.24).—A grinding machine of the disintegrator type is provided with a fan for withdrawing the ground material. The fan has curved pivoted blades which will yield without breaking when struck by a piece of uncrushable material, and are held in their working position by centrifugal force; this enables the fan to run at the same high speed as the disintegrator and to be driven by the same shaft. The disintegrator beaters are loosely mounted on pivots and are of such a shape that they will automatically change their position and expose fresh wearing surfaces every time the mill is stopped. B. M. Venables.

Filtering liquids [*e.g.*, separating oil from water]. Pirbright Co., Ltd., and J. T. Peddie (E.P. 243,107, 9.9.24).—A filter suitable for the separation of a small quantity of oil from water is constructed of cow hair and a coarse textile fibre, *e.g.*, jute, made into a felt and associated with wire or a perforated backing. B. M. Venables.

Apparatus for filtering dust-laden gases. T. Thomson and N. Nisbet (E.P. 243,117, 18.9.24).—Gases such as blast-furnace gas are filtered several times through the same moving diaphragm, the crude gas passing first through the freshly cleaned diaphragm, which is capable of retaining only the coarser particles of dust. The deposition of dust partially closes the pores of the diaphragm and makes it capable of retaining the finer dust when gas is again passed through. All the passes must be in the same direction through the diaphragm. The diaphragm preferably consists of loose material which is allowed to descend between vertical perforated walls to a cleaning apparatus at the bottom. B. M. Venables.

[Edge] filters. General Electric Co., Ltd., and L. D. Goldsmith (E.P. 243,176, 6.12.24).—In a filter of the edge or stream-line type for filtering hot gases, the impermeable sheets are made of fibrous heat-resisting material with a rough surface, *e.g.*, asbestos paper. B. M. Venables.

Treatment of liquids [*e.g.*, continuous-current washing of mineral oils]. T. A. Smith (E.P. 243,113, 13.9.24).*—A liquid is washed by successive intermixture with another liquid of different density and the mixed liquids are passed successively into a series of settlers, *a*, *a*¹, *a*². The mixers may conveniently take the form of centrifugal or other pumps, *b*, *b*¹, *b*², which also act as circulating pumps. The liquids circulate in counter-current as indicated by the arrows, and the delivery pipe, *d*, of each pump is divided, so that part of the mixed liquids passes to the settler from which the less dense liquid is derived and part to the settler from which the denser liquid is derived. The method is applicable to the refining of mineral oils. B. M. Venables.



Still. M. F. de Bajligethy (U.S.P. 1,562,690, 24.11.25. Appl., 20.6.22).—The still comprises a boiler in which a number of drums are mounted, one end of each drum projecting from the boiler and being provided with a detachable head. The inner end of each drum is provided with an inlet pipe, part of which is formed as a heating coil in a furnace setting, and the outer end of the drum is provided with a nozzle discharging into the boiler. H. Holmes.

Evaporator. H. D. MILES, Assr. to BUFFALO FOUNDRY AND MACHINE Co. (U.S.P. 1,562,713, 24.11.25. Appl., 28.1.24).—Inclined up-take tubes are mounted longitudinally within an inclined steam-chest with their ends rigidly secured in upper and lower stationary flue-sheets. The tubes communicate with an upper exhaust chamber and a lower return chamber rigidly connected to the upper and lower ends respectively of the steam-chest. The chambers also communicate through an inclined return tube disposed beneath the steam-chest. This tube is connected to one chamber rigidly, and to the other chamber in such a way that it is free to slide.

H. HOLMES.

Treating [concentrating] liquid materials. G. D. HARRIS, Assr. to INDUSTRIAL DRYER CORP. (U.S.P. 1,562,760, 24.11.25. Appl., 23.10.20).—The liquid is heated and exposed to a drying atmosphere as it flows tranquilly by gravity in a film in contact with a succession of inclined surfaces.

H. HOLMES.

Drying semi-liquid materials. G. D. HARRIS, Assr. to INDUSTRIAL DRYER CORP. (U.S.P. 1,562,761, 24.11.25. Appl., 23.10.20).—The material is delivered so as to form a film on an exposed portion of an endless carrier moving through a drying chamber, and flows through a succession of channels defined by baffles adjacent to the carrier. The channels are provided with central heating devices and with transverse baffles staggered in relation to the carrier and to the main baffles, the two series being disposed alternately adjacent to the upper and lower boundaries of the channels. Means are provided for delivering air at normal temperature to the channel adjacent to the point of discharge of the material, for circulating the air through the channels in a direction opposed to the flow of the material, and for discharging the heated air adjacent to the point of delivery of the material.

H. HOLMES.

Drying apparatus. G. D. HARRIS, Assr. to INDUSTRIAL DRYER CORP. (U.S.P. 1,562,762–3, 24.11.25. Appl., [A] 31.3.21, [B] 8.8.22).—(A) The material or object under treatment is placed on the floor of a chamber provided with an inlet and an outlet for an oxidising or drying atmosphere. Means are provided for circulating this atmosphere so that it is repeatedly directed downwards on to the material, and independent means are provided for feeding fresh air into the chamber. (B) The material is subjected to contact with a drying atmosphere as it moves through successive channels within a chamber. The atmosphere is directed downwards upon the centre of the stream of material and then flows across the material transversely of the stream.

H. HOLMES.

Drying. G. D. HARRIS, Assr. to INDUSTRIAL DRYER CORP. (U.S.P. 1,562,764, 24.11.25. Appl., 20.10.22).—A drying atmosphere is heated and subsequently cooled to promote its circulation in contact with the material to be dried. The humidity of this atmosphere is controlled in accordance with the content of surface moisture and its diffusion within the material by condensing the moisture content of the atmosphere to a definite dew-point. The atmosphere is heated to a constant temperature in the periods between its initial heating and subsequent cooling.

H. HOLMES.

Liquefaction of gases. C. E. RECORDON and J. W. HILLE, Assrs. to AIR REDUCTION Co. (U.S.P. 1,562,915, 24.11.25. Appl., 24.6.22).—The gaseous mixture is supplied to one of two chambers connected to the bottom of two sets of tubes, contained in a case filled with a refrigerating liquid. The two sets of tubes are connected at the top by a header, but the chambers at the bottoms are separated by a partition, which can expand longitudinally while remaining gas-tight; this prevents contamination of the liquid collected in the second chamber.

A. GEAKE.

Condenser. E. W. ISOM and J. E. BELL, Assrs. to SINCLAIR REFINING Co. (U.S.P., 1,563,492, 1.12.25. Appl., 19.5.21).—A tank to contain condensate and a cooler are connected so as to provide a closed circulation from the tank through the cooler and back to the tank. The vapours to be condensed are injected below the normal level of the condensate contained in the tank, and means are provided for withdrawing condensate from the tank.

H. HOLMES.

Preparing [nickel] catalytic material. M. RANEY (U.S.P. 1,563,587, 1.12.25. Appl., 20.9.24).—Nickel is alloyed with an equal weight of silicon, and the alloy is pulverised and treated with caustic soda until the silicon is converted into sodium silicate. The finely-divided catalytic material is then removed from the solution.

H. HOLMES.

Drying apparatus. FULLER-LEHIGH Co., Assees. of D. V. SHERBAN (E.P. 242,604, 19.9.25. Conv., 6.11.24).—See U.S.P. 1,558,119; B., 1926, 2.

Process of making material for filtering and decolorising. P. L. WOOSTER (Reissue 16,225, 15.12.25, of U.S.P. 1,518,289, 9.12.24. Appl., 5.8.25).—See B., 1925, 86.

Centrifugal separator. G. R. SCHUELER (U.S.P. 1,564,632, 8.12.25. Appl., 27.9.23).—See E.P. 217,065; B., 1924, 697.

Apparatus for removing material from the wall of a revolving centrifugal basket. E. VAN DER MOLEN (U.S.P. 1,565,605, 15.12.25. Appl., 16.12.24).—See E.P. 238,460; B., 1925, 790.

Means for transfer of heat. (E.P. 242,690, 29.7.24).

Atomising apparatus. E. C. MARCESCHE (E.P. 239,488, 27.11.24. Conv., 8.9.24. Addn. to 238,128).

Apparatus for separating oil from liquids. D. B. MORISON (E.P. 243,428, 25.8.24).

Apparatus for separating fuel and other oils from water. E. W. Green and H. Ogden (E.P. 243,433, 26.8.24 and 8.1.25).

Disintegrators for grinding coal and the like. F. Twort (E.P. 244,003, 19.6.25).

Gas producer and furnace for steam generators (E.P. 243,092).—See II.

Separating volatile constituents from materials. (E.P. 214,273).—See XII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Sardinian coals, especially those of the Gonnesa [Inglesias] field. R. Binaghi and D. Romoli-Venturi (Annali Chim. Appl., 1925, 15, 397—410).—The view that the Sardinian coals are lignites has prejudiced their use even in cases where they could with advantage have replaced English anthracite. Laboratory and large-scale tests show that by low-temperature distillation (Benzo-naftène process) they furnish a high-grade gas in yield equal to that obtained from the best coal and that all the by-products are commercially valuable.

W. E. Ellis.

Peat and peat problems in Sweden. S. Odén (Fuel, 1925, 4, 505—527; cf. B., 1924, 817).—The formation, constitution, and utilisation of peat is dealt with, particularly in relation with the peat resources of Sweden. The formation of peat is illustrated by sections through the more important peat bogs. The changes involved in ulmification are discussed from a comparison of analyses of peat from different depths. For the chemical characterisation of peats it is suggested that the following six groups be determined :—(1) fats, waxes, and resins by extraction with ether; (2) lignoid substances by extraction with sulphurous acid at 110°; (3) ulmins by extraction with ammonia at 110°; (4) cellulose by Schweitzer's reagent; (5) insoluble organic residue, and (6) inorganic residue. Further special groups may be determined, *e.g.*, total hexoses and pentoses produced on inversion, pentoses and methylpentoses, methoxyl, ulmic acids colorimetrically, and nitrogen. The utilisation of peat in agriculture is briefly outlined. For its utilisation as fuel the removal of the excess water is of the first importance. The simple theory of water removal from the peat capillaries by pressure leads to the relation: water removed × pressure = a constant for the same peat. This has been experimentally verified for several samples of peat. Some calculations of the economics of different drying processes are given. The yields and composition of the products of "wet carbonisation" and low-temperature carbonisation at temperatures from 150° to 400° are tabulated for a number of samples of peat. For ammonia recovery, distillation in steam at 700° gives the best yield, equivalent to about 55% of the nitrogen in the peat. Some experiments were made on heating peat in an autoclave with sodium hydroxide at 365°. The product was distilled, first directly, then with superheated steam, and yielded a gas (mainly hydrogen), methyl alcohol, acetone, and light and heavy oils. In experiments on the anaerobic fermentation of peat, a loss of pentosans and hexosans was observed, the relative loss of the latter being the greater.

A. B. Manning.

Possible use of Philippine coals for liquid fuel. V. G. Lava (Philippine J. Sci., 1925, 28, 193—204).—When an emulsion of crude oil in water stabilised by soap is mixed with coal (200-mesh) a stable liquid fuel is obtained. Potassium soaps produce greater stability than sodium soaps. The greatest stability was obtained with suspensions containing 7·7 and 8·7% of water. Higher calorific value, but lesser stability were given with 4·2 and 4·8% of water. Differences in the stability of suspensions were noted with variation in composition of the coal, the stability being greater with coals having a low content of volatile combustible matter. The calorific value of the suspensions varied from 8670 cals. with 6% of water to 8720 cals. with 4·8% of water. The coal content of the suspensions was approximately constant at 46%, and the content of crude oil varied from 46·2 to 48·5%.

W. S. Patterson.

Test of the vertical [gas] retorts with a 24-hr. [carbonising] period at the Hanover gasworks. F. Kleemann (Gas- u. Wasserfach, 1925, 68, 747—748).—The results of a three-day test on a vertical retort system erected by the Dessauer Vertikalofen-Ges. are tabulated. The system consists of ten vertical chamber retorts, of which, however, only eight were in operation during the test. Each retort consists of six chambers, each of five tons capacity, and is provided with two generators and recuperator. Each chamber is provided with two steam inlet ports. The carbonisation period was 24 hrs., and the charge was steamed for 9 hrs. A Ruhr coal (Ewald) with 2·4% of moisture, 29·2% of volatile matter, and 8·8% of ash, was used. The average temperature in the chambers was 1040°. The average gas yield was 41·9 $m.^3$, of gross calorific value 4625 cal., per 1000 kg. of coal. The average coke consumption was 13·8 kg. (dry) per 100 kg. of coal.

A. B. Manning.

Mechanism of combustion in the Bunsen cone. E. W. Rembert and R. T. Haslam (Ind. Eng. Chem., 1925, 17, 1233—1236).—The rate of combustion of a mixture of fuel gas and air, burning as a Bunsen cone in a small furnace, was determined by analysis of the flue gases, sampled at a point 4¼ in. above the port. The rate of combustion was found to be independent of the temperature of the furnace walls between 110° and 1100°. This is verified by direct calculation of the change in the radiation heat losses from the flame. On increasing the air : gas ratio, complete combustion was attained a short distance above the port with only 5% excess air. It is concluded that the rate of gaseous combustion in furnaces is controlled by the rate of mixing of secondary air with the combustible gases.

The effect of varying port velocity, port diameter, and the air : gas ratio on the height of the Bunsen cone was observed. The cone height increased with gas velocity up to a point, and was then practically constant. The cone height increased with port diameter, but in some cases passed through a maximum. The cone height decreased with increase of the air : gas ratio. A. B. MANNING.

Factors influencing length of a gas flame burning in secondary air. E. W. REMBERT and R. T. HASLAM (Ind. Eng. Chem., 1925, 17, 1236—1238; cf. preceding abstract).—The length of a gas flame burning in free space can be represented by an equation of the form $L=K \log u-B \log D-E$, where u is the port velocity, D is the port diameter, and K, B, and E depend on the primary air : gas ratio. If a constant quantity of fuel gas is supplied to a burner per unit time, and the amount of primary air mixed with the gas increased from zero, the length of the flame decreases. The ratio of secondary air to gas has practically no effect upon the length of the flame, provided this ratio does not fall below that required for the combustion of 75% of the fuel gas. A. B. MANNING.

Factors affecting utility of secondary air in gaseous combustion. E. W. REMBERT and R. T. HASLAM (Ind. Eng. Chem., 1925, 17, 1238—1240; cf. preceding abstracts).—The flue gases from a fuel gas flame, burning in a measured supply of secondary air only, have been carefully sampled and analysed. Curves showing the variation of air utilised for combustion with air supplied, port velocity, and port diameter, have been plotted. The total amount of air supplied materially affects the fraction thereof used for combustion. The fraction, air used $\div$ air supplied, passes through a maximum as the supplied air is increased. The position of the maximum depends on the port velocity and diameter. The fraction of supplied air that is utilised for combustion increases with both port velocity and diameter, owing probably to an increased rate of mixing. Over the range investigated the log. of the air supplied is practically a linear function of the air utilised.

A. B. MANNING.

Relative rates of combustion of constituents of city gas burning in secondary air. E. W. REMBERT and R. T. HASLAM (Ind. Eng. Chem., 1925, 17, 1240—1242; cf. preceding abstracts).—From the analyses of the flue gases from city gas burning in varying amounts of secondary air, relative mass action constants have been deduced giving the ratios of the rates of combustion of the different components of the gas. The combustion of methane and ethylene proceeds according to the equations $CH_4+O_2=CO+H_2+H_2O$ and $C_2H_4+O_2=2CO+2H_2$, where $k_{C_2H_4}/k_{CH_4}=1{\cdot}1$. The relative rates of combustion of hydrogen and carbon monoxide, burning simultaneously in the presence of small quantities of methane and ethylene, follow an expression of the type $1/H_2=(k_{H_2}/k_{CO})1/CO+c$, where $k_{H_2}/k_{CO}=4{\cdot}9$ (approx.). Similar expressions hold for the simultaneous combustion of hydrogen and methane, $k_{CH_4}/k_{H_2}=3{\cdot}0$, and of carbon monoxide and methane, $k_{CH_4}/k_{CO_2}=14{\cdot}5$. A. B. MANNING.

Effects of knock inducers and suppressors upon gaseous ionisation. G. L. CLARK, E. W. BRUGMANN, and W. C. THEE (Ind. Eng. Chem., 1925, 17, 1226—1229).—The experiments of Wendt and Grimm on the effect of knock suppressors and inducers on the rate of re-combination of gaseous ions (B., 1924, 856) have been repeated under carefully controlled conditions. A constant current of air, air+benzene, air+benzene+butyl nitrite, or air+benzene+lead tetraethyl, was ionised by exposure to monochromatic X-rays ($K\alpha$ of molybdenum), and its ionisation was measured at two points about 20 cm. apart in a secondary ionisation chamber. The ratio of the ionisation currents at the two points gives a measure of the relative rates of re-combination of the ions. The mixtures were obtained by passing the air current over benzene, a 2% solution of butyl nitrite in benzene, or a 3·4% solution of lead tetraethyl in benzene, respectively. The presence of butyl nitrite slightly increased the total ionisation of the benzene–air mixture, and slightly decreased the rate of re-combination. This is in accord with the theory of Wendt and Grimm. The presence of lead tetraethyl gave a still larger total ionisation, and a greater decrease in the rate of re-combination. This accords neither with the theory nor with the results of Wendt and Grimm. The above results were confirmed by a second series of experiments using a specially designed and very sensitive ionisation chamber and quadrant electrometer. The theories that have been suggested to explain the action of knock suppressors are summarised. A. B. MANNING.

Chemical equilibrium in gases exhausted by gasoline engines. W. G. LOVELL and T. A. BOYD (Ind. Eng. Chem., 1925, 17, 1216—1219).—A consideration of the data of Fieldner and his co-workers on the composition of the gases exhausted by automobile engines (B., 1921, 111 A; 1922, 622 A) shows that the value of the ratio $K=(CO)(H_2O)/(CO_2)(H_2)$ lies within narrow limits, 3·0—4·0, over the whole range of conditions of mixture ratio and other variables employed. It is concluded that in the ordinary automobile engine there is substantial equilibrium of the reaction $CO_2+H_2 \rightleftharpoons CO+H_2O$. The value of K corresponds to the equilibrium value for this reaction at temperatures of 1350—1550°. Measurements of the maximum temperatures occurring in the cylinders of gasoline engines by other investigators give values of the same order, 1500—1800°. Over 95% of the carbon in the fuel was burnt to carbon monoxide plus carbon dioxide for the range of variables over which the engines ran satisfactorily. The relative amounts of hydrogen burnt to water and carbon burnt to carbon monoxide confirm the hydroxylation theory of the combustion of hydrocarbons, and disprove the old belief that the reason for the

maxima of the power curves of gasoline engines occurring at about 85% of theoretical air was due to a preferential burning of hydrogen.

A. B. MANNING.

Vacuum assay distillation test [for mineral oils]. A. G. PETERKIN and S. W. FERRIS (Ind. Eng. Chem., 1925, **17**, 1248—1249; cf. U.S. Bur. Mines, Bull. 207, 1922).—A simplified vacuum distillation apparatus for carrying out routine tests of medium and heavy oils is described. The apparatus includes a device for maintaining a constant pressure of 10 mm. 100 c.c. of the oil are distilled from a 250-c.c. Claisen flask under the reduced pressure, into a 100-c.c. measuring cylinder, the procedure being similar to that of the Engler distillation test. A. B. MANNING.

Relation between boiling point and some other properties of petroleum products. J. B. HILL and S. W. FERRIS (Ind. Eng. Chem., 1925, **17**, 1250—1252; cf. preceding abstract).—The relations between some physical properties of a number of close-cut fractions from various crude petroleums, and the boiling ranges of the fractions as determined by the vacuum assay distillation test, have been studied. Five typical crude petroleums were examined, ranging from a Pennsylvania crude to a highly naphthenic Gulf Coast crude. The b.p. of the fractions, as given by the 50% point on the vacuum assay distillation, were plotted against viscosity, temperature coefficient of viscosity, specific gravity, and refractive index. For fractions of the same b.p., these properties all increase as the crude oil varies from paraffinic to naphthenic. The relations observed can be used as a good indication of the source of an unknown oil. A. B. MANNING.

Phosphorescent flame of carbon disulphide. DIXON and HIGGINS.—See A., 1926, 10.

Union of carbon monoxide and oxygen in contact with a gold surface. BONE and ANDREW. —See A., 1926, 33.

Hydrogenation of ethylene. PEASE and STEWART. —See A., 1926, 43.

PATENTS.

Production of water-gas from solid fuel. J. RUDE (E.P. 235,007, 18.6.24).—In the production of water-gas in externally-heated retorts, the steam is admitted at various points and levels, the gases being withdrawn either through pipes at different levels passing through the wall of the retort to a main, or through a central outlet pipe. The gases may be superheated and used for preheating the fuel as described in E.P. 204,718 (B., 1924, 663). The steam may be admitted at different levels alternately, in which case the zones not receiving steam are reheated preparatory to being again steamed.

Utilisation of the waste gases from water-gas generators. C. MARISCHKA (E.P. 236,579, 3.7.25. Conv., 5.7.24).—The heat in the waste "blow" gases from water-gas generators having a steam-generating jacket is utilised by passing the gases, either directly or through a combustion chamber, where they are burnt with air, into a jacket chamber surrounding the generator boiler. This arrangement ensures a better thermal efficiency with smaller costs and less floor space than the use of a separate waste-heat boiler. The combustion chamber may also serve as a heat accumulator, the water-gas generated in the second period being led through it and thence through the jacket chamber of the boiler; or it may be used for superheating the steam supplied to the generator. The heating surface in the jacket chamber is increased by the addition of a number of vertical water tubes to the boiler. In a modification the jacket chamber is lined with heat-storing material, and itself serves as a combustion chamber.

A. B. MANNING.

Combined gas producer and furnace for use with steam generators. T. R. WOLLASTON (E.P. 243,092, 28.8.24 and 26.2.25).—A gas producer is combined with the furnace space or fire-box of a vertical or horizontal type of boiler, the normal grate of the fire-box being omitted, and communication being made between the two by one or more "bottle-neck" apertures, through which the fuel, fed in at the ordinary fire-door, passes down into the producer. The "bottle-neck" may be provided with a door or valve. A fan supplies primary air to the producer, and secondary air to the furnace chamber. The producer is cased and lined, and is provided with annular spiral passages, in which the secondary air is preheated. Separate additional communications other than the "bottle-neck" may be provided between the producer and the furnace chamber to act as gas outlets. The piping from the fan to the producer or other parts involved may pass through the smoke-box of the boiler, and the air supply be thus further preheated. A. B. MANNING.

Vertical or inclined gas-making retorts. T. R. WOLLASTON (E.P. 243,169, 1.12.24; cf. E.P. 113,856, 207,700, and 209,352; B., 1918, 233 A; 1924, 209).—A composite gas retort comprises an upper vertical portion, in which the fuel undergoes a pre-coking process, and a lower portion, either vertical or inclined, in which the coking proper takes place. The gases from the lower portion pass up and over the fuel or coal in the upper portion, which is thereby heated. If required, the upper portion may, like the lower, be mounted in an ordinary setting with the usual flues or combustion chambers, and so be further suitably heated. The time of complete coking is shortened by this process, and the difficulties arising from excessive caking or swelling are avoided.

A. B. MANNING.

Coking process [for pitch]. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,563,595, 1.12.25. Appl., 18.7.19).—A low-ash coke is produced from liquefiable bituminous material by heating a proportion of the material to expel part of the volatile matter, mixing the semi-coke so formed with a proportion of the original material, and completely coking the mixture. A. B. MANNING.

Utilisation of grape residues. F. DUPLAN (F.P. 590,739, 15.2.24).—By dry distillation of the residues from the manufacture of wine, ethyl and methyl alcohols, acetic acid, and tar are obtained as well as combustible gases and a highly absorbent type of carbon. The tar is similar to that obtained by distillation of peat or brown coal and contains aromatic hydrocarbons and oils of high boiling point.
C. T. GIMINGHAM.

Purifying materials for acetylene and other gases. CHEM. FABR. GRIESHEIM-ELEKTRON, and A. HERMANN (E.P. 243,607, 24.4.25).—See U.S.P. 1,551,878; B., 1925, 873. To render the material porous, the mass, prior to solidification, is mixed with a small quantity of a substance, *e.g.*, finely divided aluminium or magnesium, which will react to generate gas.

Dehydrating [oil] emulsions by externally-charged particles. W. MEREDITH, Assr. to PETROLEUM RECTIFYING Co. (U.S.P. 1,562,712, 24.11.25. Appl., 14.2.21).—Conducting particles (*e.g.*, of water) are separated from an insulating medium (*e.g.*, oil) by introducing into the medium other particles, electrically charged, and subjecting the mixture to an electric stress. H. HOLMES.

Process of forming activated carbon. G. W. WALLACE (U.S.P. 1,565,129, 8.12.25. Appl., 15.5.25). —See E.P. 234,161; B., 1925, 661.

Cracking hydrocarbons. F. G. NIECE, Assr. to INTERNATIONAL HOLDING Co. (U.S.P. 1,565,326–7, 15.12.25. Appl., 7.7 and 13.7.21).—See E.P. 185,632; B., 1922, 850 A.

Bottom grids for gas purifiers. S. E. SPENCER (E.P. 243,867, 22.10.24 and 11.6.25).

Treatment of mineral oils. (E.P. 243,113.)—See I.

Mixtures of nitrogen and hydrogen for ammonia synthesis (E.P. 243,122).—See VII.

III.—TAR AND TAR PRODUCTS.

Distribution of pyridine between water and benzene. WOODMAN and CORBET.—See A., 1926, 19.

Reduction of organic compounds containing oxygen [cresols] by active carbon. STADNIKOV, GAWRILOV, and WINOGRADOV.—See A., 1926, 60.

PATENT.

Treating wood-tar oil. A. E. MAZE, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,558,446, 20.10.25 Appl., 27.3.24).—A distillate, boiling up to 130°, obtained from wood-tar oil is agitated in the presence of bleaching powder. The product has an improved odour and is a good solvent for nitrocellulose.
A. J. HALL.

IV.—DYESTUFFS AND INTERMEDIATES.

Comparative study of azo dyes made with H-acid and acetyl-H-acid. W. W. LEWERS and A. LOWY (Ind. Eng. Chem., 1925, 17, 1289—1290).—The hypsochromic effect due to the introduction of an acetyl group has been studied by the coupling of 33 diazotised compounds with H-acid and with acetyl-H-acid respectively. Tables are given for each of the two series of dyes which show the % yields, the appearance of the dyes as powders, the shades produced on wool, the colours of the aqueous solutions, and the colour changes of aqueous solutions when acted on by hydrochloric and sulphuric acids and sodium hydroxide respectively.
A. COULTHARD.

Cyanine dyes. MILLS and RAPER.—See A., 1926, 77.

Diazotisation of picramide. BLANGEY.—See A., 1926, 62.

Solubility and retention of water of crystallisation in salts of aromatic sulphonic acids. EPHRAIM and SEGER.—See A., 1926, 18.

PATENTS.

Manufacture of azo colouring matters dyeing wool fast to milling. BRITISH DYESTUFFS CORP., LTD., J. BADDILEY, J. HILL, and A. RILEY (E.P. 243,115, 17.9.24).—Azo dyestuffs suitable for dyeing wool in yellow to brown shades fast to milling are obtained by coupling a diazotisable compound, prepared by the action of at least 1 mol. of formaldehyde on 1 mol. of the hydrochloride of an aromatic amine, with a sulphonated pyrazolone, naphthylamine, or naphthol, or an aminonaphtholsulphonic acid. For example, 90 pts. of formaldehyde solution (containing 38·7 pts. of CH_2O in 100 c.c.) are added to a solution at 20° containing 93 pts. of aniline, 106 pts. of hydrochloric acid (*d* 1·18), and 200 pts. of water, whereby the temperature of the mixture rises to 60°; after 24 hrs. the anhydro-base $(CH_2.C_6H_4.NH)_n$ produced is precipitated by the addition of an excess of alkali filtered off, washed, and dried. A 10% solution of the anhydro-base in dilute hydrochloric acid is diazotised at 5° with 70 pts. of sodium nitrite and poured into a cold solution containing 254 pts. of 1-*p*-sulphophenyl-3-methyl-5-pyrazolone, 3000 pts. of water, 40 pts. of caustic soda, and 160 pts. of sodium carbonate. The resulting dyestuff is yellow. Suitable anhydro-bases are prepared from *o*-anisidine, *m*-nitroaniline, and β-naphthylamine, and suitable compounds for coupling include 1-(2 : 5-dichloro-4-sulpho)phenyl-3-methyl-5-pyrazolone, 2-naphthol-6-sulphonic acid, 2 : 8-aminonaphthol-6-sulphonic acid, and 2 : 7-naphthylaminesulphonic acid.
A. J. HALL.

Preparation of 5(?)-nitro-3-amino-6-hydroxyacetophenone. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING, Assees. of K. STREITWOLF and A. FEHRLE (G.P. 417,444, 8.1.24).—3-Acetamido-6-hydroxyacetophenone is added to concentrated

sulphuric acid with cooling, and a mixture of concentrated sulphuric and nitric acids (*d* 1·38) is then added, drop by drop, at —10° to 0°. On pouring on to ice there is obtained, in 92·4% yield, yellow 5 (?) - nitro - 3 - acetamido - 6 - hydroxyacetophenone, which on crystallisation from alcohol, hydrolysis with hydrochloric acid, and precipitation with ammonia yields orange-yellow 5(?)-nitro-3-amino-6-hydroxyacetophenone, m.p. 142° (decomp.). The latter after diazotisation couples with 1-amino-8-hydroxynaphthalene-3 : 6-disulphonic acid (violet) and β-naphthol (red). B. FULLMAN.

Preparation of tetrabenzoylperylene. H. PEREIRA, and COMP. NAT. DE MATIÈRES COLORANTES ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES, ETABL. KUHLMANN (F.P. 591,271, 31.12.24. Conv., 10.4.24).—1 pt. of perylene, at least 2 pts. of benzoyl chloride, and aluminium chloride are heated in carbon disulphide, *e.g.*, for 6 hrs. to boiling, the solvent is distilled off, and the resulting aluminium chloride double compound decomposed with hydrochloric acid. Tetrabenzoylperylene, dark yellow prisms, m.p. about 320°, is soluble in high-boiling organic solvents. Its concentrated sulphuric acid solution is bluish-green, with strong red fluorescence. B. FULLMAN.

Manufacture of *o*-[hydr]oxyazo-dyestuffs. AKT.-GES. FÜR ANILIN-FABR. (E.P. 232,629, 18.4.25. Conv., 19.4.24).—See U.S.P. 1,556,329; B., 1926, 7.

Manufacture of benzanthrone derivatives. BADISCHE ANILIN- U. SODA-FABR., Assees. of A. LÜTTRINGHAUS, H. WOLFF, and H. NERESHEIMER (U.S.P. 1,564,423, 8.12.25. Appl., 26.4.23).—See E.P. 203,533 ; J., 1923, 1064 A.

Condensation products [dyestuffs] of the anthraquinone series. G. KRÄNZLEIN and R. SEDLMAYR, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,564,584, 8.12.25. Appl. 27.9.23).—See E.P. 205,502 ; B., 1924, 627.

Bluish sulphurised indophenolbenzidine dye. SOC. OF CHEM. IND. IN BASLE, Assees. of L. HAAS and E. REBER (U.S.P. 1,565,736, 15.12.25. Appl., 7.5.23).—See E.P. 199,360 ; B., 1924, 9.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Testing of wood pulps with the large size Lampen ball mill. R. SIEBER (Ver. Zellstoff- u. Papier-Chem., Hauptversamml., 1924, 175—176; cf. B., 1925, 540).—The Lampen mill, consisting of a single heavy bronze ball in a revolving casing, has proved more satisfactory for beating test-samples than the more usual type of porcelain ball-mill. In order to avoid "chattering" the mill should revolve at not more than 200 revs. per min. The single ball may require a longer time to produce the desired result than several balls, but the uniformity and comparative accuracy of the beating should be more certain. The beating result would appear to depend not so much on the force of the strokes as upon their number or frequency, and this is controlled by the single ball. The substitution of metal for porcelain has proved very satisfactory. The sheet of paper is produced on a mould connected with a water suction pump and is pressed up, after couching, in a copying press. Uniformity of treatment is essential. A drying arrangement is used consisting of a steam-heated horizontal cylinder with thermometers at inlet and outlet. The cylinder is provided with a felt which is attached to the cylinder at one end and weighted with an iron rod at the other end, so that weights may be applied to hold the sheet of paper stretched between the felt and the cylinder. J. F. BRIGGS.

Disintegration of fibrous raw materials by means of nitric acid. P. KRAIS (Papier-Fabr., 1925, 23, 797—799 ; cf. G.P. 391,713 and 395,191 ; B., 1925, 68, 626).—The use of nitric acid is economically sound where this is cheap, *e.g.*, in South America and in places where cheap electric power is available. A. GEAKE.

Colloidal properties of sericin. BROSSA.—See A., 1926, 22.

Decomposition of cellulose by fungi. HEUKELEKIAN and WAKSMAN.—See XVI.

PATENTS.

Paper-making machinery. R. R. BLIGHT (E.P. 243,640, 2.7.25).

Organic thiocarbonates (F.P. 563,214).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Use of sodium silicate in printing [textile fabrics] with vat dyestuffs. LA MANUFACTURE E. ZUNDEL, and L. LANTZ (Sealed Note 2088, 22.5.11. Bull. Soc. Ind. Mulhouse, 1925, **91**, 559—560. Report by P. BINDER, *ibid.*, 560—561).—Sodium or potassium carbonate is replaced by sodium silicate in printing pastes containing vat dyestuffs since this substitute may be easily freed from suspended grit and it also assists the preparation of highly thickened printing pastes. Satisfactory results are obtained by means of a printing paste prepared by heating a mixture containing 200 pts. of prepared indigo paste, 125 pts. of gum water, 50 pts. of sodium silicate of 35° B. (*d* 1·32), 75 pts. of caustic soda of 38° B. (*d* 1·36), and 50 pts. of a mixture of sodium hyposulphite and glycerin, at 50° until the dyestuff is completely reduced and then adding 170 pts. of gum water, 300 pts. of sodium silicate (*d* 1·32), and 30 pts. of Rongalite C; the prepared indigo paste contains 200 pts. of Indigo 2 B (M.L.B.) powder, 200 pts. of sodium silicate (*d* 1·32), and 600 pts. of gum water. Binder reports favourably on the process but points out that dilute solutions of sodium silicate tend to become gelatinous. A. J. HALL.

[Production of] scroop effects on cotton. C. SUNDER (Bull. Soc. Ind. Mulhouse, 1925, **91**, 561—562).—A scroop finish is imparted to cotton materials by impregnation with a solution containing

per 100 litres, 1 litre of an emulsion prepared by agitating a mixture of 180 pts. of coconut oil, 10 pts. of oleic acid, 20 pts. of caustic soda of 10° B. (*d* 1·075), and 400 pts. of water. Sodium soaps are unfavourable and triglycerides are favourable to the production of scroop effects. When the fabric being treated contains soap, an ammonium salt should be added to the emulsion described above, but the shade of the fabric may then be affected; Alizarin Pink shades are affected, and although ultramarine is scarcely affected it is preferably replaced by Indanthrene Blue. A. J. Hall.

Patents.

Dyeing, printing or stencilling cellulose acetate. British Celanese, Ltd., and G. H. Ellis (E.P. 242,393, 19.9.24. Addn. to 219,349; B., 1924, 906).—Solubilised dyestuffs suitable for dyeing cellulose acetate are prepared by treatment of dyestuffs previously described (cf. E.P. 219,349, *loc. cit.*, also E.P. 224,681, 227,183, and 237,943, B., 1925, 39, 204, 801) with sulphoaromatic fatty acids, *e.g.*, sulphobenzene-stearic acid (Twitchell reagent), sulphophenol-stearic acid and sulphonaphthalene-stearic acid, which act as solubilising agents (cf. E.P. 219,349, *loc. cit.*, and E.P. 224,925, B., 1925, 39). A suitable solubilising agent is prepared by adding a cold paste containing 25 kg. of naphthalene (or benzene) and 25 kg. of oleic acid to 100 kg. of 20% oleum at 40°, the temperature being then raised to 100° and maintained for 3 hrs. The product is poured into 250 litres of water containing 50 kg. of sodium chloride and the upper layer is separated and purified. A. J. Hall.

Centrifugal machine for treating textiles with liquid and gases. H. Wolfer, Assr. to Obermaier u. Co. (U.S.P. 1,564,503, 8.12.25. Appl., 8.12.24).—See E.P. 226,527; B., 1925, 239.

Textile fabric dyeing machines. E. Cadgene (E.P. 242,936, 2.3.25. Conv., 13.11.24).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

[Combustion temperatures of] pyrites and zinc-blende. J. Baumann (Chem.-Ztg., 1925, 49, 1061).—The maximum temperature attainable by the combustion of pyrites, assumed to contain 48% of sulphur as FeS_2 and 10% of silica, in the chamber process of sulphuric acid manufacture is calculated to be 1216°. That of zinc-blende having 28·4% of sulphur and 14% of silica is calculated as 1234°. The exit gases from the chamber plant are assumed to contain 6·0—6·5% of oxygen. These figures are maxima, no allowance having been made for losses due to radiation, incomplete combustion, or secondary reactions. C. Irwin.

Influence of water vapour and hydrogen chloride on the velocity of decomposition of ammonia. A. Schmidt (Z. angew. Chem., 1925, 38, 1146—1154).—Experiments were made with a current of pure ammonia flowing at constant pressure through a 100-c.c. porcelain bulb with capillary inlet and outlet and maintained at a known temperature, the volume of nitrogen and hydrogen being measured. The undecomposed ammonia was determined by titration. It was found that at all temperatures from 700° to 1000° the decomposition velocity of dry ammonia increased steadily over a period of many days to a figure many times the initial one. The inner wall of a used porcelain bulb was found to be coated with a grey substance which was proved to be metallic iron and in a particular case amounted to 0·04 mg. per sq. cm. This iron catalyses the decomposition of ammonia, causing the progressive increase in the decomposition velocity. It appears that iron passes from the interior of the porcelain to the surface. The deposit is destroyed by heating with ammonia gas saturated with aqueous vapour. Quantitative tests showed that small proportions of water vapour had no protective effect. The decomposition velocity began to decrease when the concentration was such as to reverse the equation, $Fe_3O_4+4H_2=3Fe+4H_2O$. To determine the effect of hydrogen chloride the porcelain surface was replaced by broken coke. The catalytic effect of separated iron on dry ammonia gas was in this case still more marked. Treatment with hydrogen chloride removed the iron on the surface of the coke and caused a decrease in the decomposition of ammonia when this was first re-admitted. The velocity of decomposition soon rose again. Longer treatment with hydrogen chloride in which the greater part of the iron content of the coke was distilled off as ferric chloride produced more permanent results. C. Irwin.

Assay of potassium chlorate. A comparison of methods. Lehn and Fink, Inc. (Ind. Eng. Chem., 1925, 17, 1277—1278).—No method involving reduction to potassium chloride with subsequent determination of the chloride was found altogether satisfactory. Standard titanous chloride solution may be used for reduction if a standard potassium chlorate of known purity is kept for standardisation, and this method is suitable for routine control analyses. Where potassium permanganate is used after reduction by excess of ferrous salt, it is important to use sodium oxalate of known purity for standardisation of the permanganate. The Frankford Arsenal method in which ferrous ammonium sulphate is used for reduction was found the most satisfactory and accurate, and better than the U.S.P. method in which a more concentrated solution (of ferrous sulphate) is used for reduction, and the solution is titrated cold. D. G. Hewer.

Oxidation of chrome iron ore in briquettes [for preparation of chromates]. P. M. Lukianov (Russian J. Chem. Ind., 1924, 1, [1], 11—13).—The conversion of chrome iron ore into sodium or potassium chromate is facilitated by making the ore (152 pts.) into briquettes with soda ash (82), quicklime (135), and water (82), prior to effecting the oxidation by roasting. With the above mixture, the degree to which the Cr_2O_3 undergoes oxidation to

CrO_3 amounts to 90% or even more in the interior of the briquettes. Addition of straw and sawdust, although it increases the porosity of the mass, lowers the extent of the oxidation; similar lowering is caused by addition of ferric oxide in the form of burnt pyrites. The use of these briquettes renders the roasting process automatic and entirely independent of skilled or experienced workmen and renders unnecessary the heavy labour required if the mixture is roasted in a finely divided form. T. H. Pope.

Heat of reaction in the formation of calcium cyanamide [from calcium carbide]. H. H. Franck and F. Hochwald (Z. Elektrochem., 1925, **31**, 581—589).—The heat evolved in the exothermic reaction $CaC_2+N_2=CaCN_2+C$ has been measured by two distinct methods. First, the heat of combustion of pure calcium cyanamide was found to be $163{\cdot}1\pm0{\cdot}4$ Cal., whence its heat of formation is $84{\cdot}0\pm0{\cdot}75$ Cal. From this and the heat of formation of calcium carbide the value $70{\cdot}5\pm2{\cdot}6$ Cal. is calculated for the heat of reaction in converting the carbide into cyanamide. By a second method the heat of reaction was directly measured in a combustion bomb charged with pure compressed nitrogen. To effect the heating-up of the carbide, the latter was packed in a small quartz tube wound with platinum wire, the whole being suspended within the bomb. A value of 72 ± 2 Cal. at constant volume was thus found. At 1100° the heat of reaction is calculated to be 67·07 cal. In the case of magnesium cyanamide the heat of formation is found 61 Cal. by the first method. W. A. Caspari.

Method of obtaining iodine, based on electrolysis in alkaline solution. L. Pisarshevski and S. Telni (Russian J. Chem. Ind., 1924, **1**, [1], 13—15).—Electrolysis of a neutral or alkaline solution of an iodide proceeds in accordance with the equation, $6I+6OH'\rightarrow5I'+IO_3'+3H_2O$, provided that the electrolyte is kept sufficiently well mixed. The solution tends to become richer in iodate, but if, at the moment when one-sixth of the total iodine in the solution exists as iodate, the electrolysis is discontinued and the electrolyte is removed and acidified, the whole of the iodine is precipitated: $6H^{\cdot}+5I'+IO_3'=3I_2+3H_2O$. Addition of a small proportion of alkali during the electrolysis is advisable in order to neutralise any acidity developing at the anode in consequence of imperfect mixing. The crude iodine obtained in this way has the composition: I, 87%; water, 12%; non-volatile residue, 0·8%; S and Cl, traces. The current efficiency amounts to 50%, the preparation of 1 kg. of iodine requiring the expenditure of 1·7 kw.-hrs. and 0·228 litre of sulphuric acid, *d* 1·83. T. H. Pope.

See also A., 1926, 16, **Compression and decomposition of nitric oxide** (Briner, Biedermann, and Rothen). 17, **Absorption of gaseous hydrogen chloride by sulphuric acid** (Cupr). 24, **Behaviour of silica gel towards alkalis and salts** (Patrick and Barclay); **Effect of temperature of formation on physical character of hydrous aluminium oxide** (Yoe). 27, **Thermal decomposition of sulphates** (Marchal). 32, **Thermal decomposition of nitrogen pentoxide at low pressures** (Hirst and Rideal). 34, **Isomeric hydrogels of aluminium hydroxide** (Willstätter, Kraut, and Erbacher). 35, **Aluminium hydroxide gel of formula AlO.OH** (Willstätter, Kraut, and Erbacher). 36, **Silicic acid** (Willstätter, Kraut, and Lobinger); **Bismuth nitrates** (Picon). 37, **Attack of hydrogen chloride and ammonium halide on metals** (Hofmann and Hartmann). 38, **Argentometric titration of iodides** (Kolthoff); **Volumetric determination of soluble sulphates** (Atkinson). 39, **Separation of silver iodide, bromide, and chloride** (Britton). 40, **Determining ferrous oxide in insoluble silicates** (Hackl).

Analysis of products rich in aluminium. Schürmann and Böhme.—See VIII.

Direct production of zinc oxide from sulphide ores. Waring.—See X.

Patents.

Absorbing hydrochloric acid gas. J. E. Egleson, Assr. to Gen. Chemical Co. (U.S.P. 1,563,732, 1.12.25. Appl., 7.9.22).—Hydrochloric acid gas and an absorbing liquid travel together through an externally cooled tube; a portion of the solution thus obtained is diluted and used for further absorption. A. Geake.

Producing carbon and hydrocyanic acid. R. W. Poindexter, Assr. to California Cyanide Co. (U.S.P. 1,562,914, 24.11.25. Appl., 4.12.22).—Carbon and hydrocyanic acid are produced simultaneously by rapidly heating a mixture of a hydrocarbon and ammonia by passage through an externally heated zone. A. Geake.

Production of silicic acid. N. L. Collins (U.S.P. 1,562,940, 24.11.25. Appl., 27.3.24).—Pure silicic acid is obtained by electrolysing an aqueous solution of sodium silicate between a rotating anode and a mercury cathode. A. Geake.

Decomposition of alkali chlorides by steam in the presence of silicate. J. Kersten (E.P. 243,104, 8.9.24).—The known process for the decomposition of alkali chloride by steam in presence of an alkali silicate containing excess silica is improved by the addition of carbon, as graphite or charcoal, to the mass. Air may be injected as well as steam to burn a part of this carbon, or the furnace may be externally heated. In either case the action of the steam on the carbon yields hydrogen, which decomposes the chloride more readily than steam alone. Three types of furnace are described, one of the "converter" type connected with separate melting pans for the raw materials, a similar one in which the melting is carried out in an expansion formed in the side of the converter, and a continuous furnace with external melting chamber and an overflow arrangement for removal of the finished product. C. Irwin.

Production of mixtures of nitrogen and hydrogen for ammonia synthesis. H. A. HUMPHREY, and SYNTHETIC AMMONIA AND NITRATES, LTD. (E.P. 243,122, 24.9.24).—If the hydrogen required for ammonia synthesis is derived from producer gas and especially when the nitrogen–hydrogen mixture is repeatedly circulated, inconvenience arises from the accumulation of methane in the gas. The maintenance of a temperature of 1300° in the producer practically eliminates the production of methane, and this can be attained either by the use of powdered fuel, or when lump fuel fed from below is used, by strongly preheating the air and steam injected. If air enriched with the required amount of oxygen can be used the correct nitrogen : hydrogen ratio in the purified gas can be obtained without further adjustment. In other cases an addition of hydrogen or of water-gas is necessary. C. IRWIN.

Method of producing pure alumina. R. JACOBSSON (E.P. 243,183, 16.12.24. Addn. to 221,209; B., 1925, 758).—The sulphuric acid used for dissolving the raw material in the method previously described need not be of greater concentration than *d* 1·3, but in this case the solution must be filtered and concentrated. The final removal of iron from the alumina is carried out by reducing the former to the metallic state and then heating to above 300° in a current of chlorine or of dry hydrogen chloride free from oxygen. This treatment is carried on in presence of the chlorides of either aluminium, chromium, tin, or carbon, or these substances may be added in the elementary state. C. IRWIN.

Aluminium chloride process. H. T. LEA and C. W. HUMPHREY (U.S.P. 1,558,897, 27.10.25. Appl., 20.6.23).—Aluminium sulphate is heated until it decomposes into aluminium oxide, sulphur trioxide, sulphur dioxide, and oxygen, and the last two gases are caused to form sulphur trioxide by passing over a catalyst. The sulphur trioxide is converted into sulphuric acid which is treated with sodium chloride to give hydrochloric acid, and the latter is converted into chlorine. The aluminium oxide obtained in the first stage of the process is mixed with carbon and treated with the chlorine to produce aluminium chloride and carbon monoxide. Alternatively, the sulphur dioxide may be caused to act directly on sodium chloride in presence of oxygen and steam to obtain hydrochloric acid. T. S. WHEELER.

Preparation of translucent rod-shaped bodies [incandescence bodies for electric lamps, artificial gems] from powdered material. GENERAL ELECTRIC CO., Assees. of PATENT TREUHAND GES. FÜR ELEKTRISCHE GLÜHLAMPEN M.B.H. (E.P. 243,251, 27.5.25. Conv., 16.12.24).—Powdered material, *e.g.*, aluminium oxide or other oxide or fluoride, is projected by an upwardly-directed blowpipe flame on to a vertical fireproof support, *e.g.*, of refractory ceramic material, and the flame is adjusted so that globules from the stream of molten material form continuously on the lower end of the support. J. S. G. THOMAS.

Method of making arsenates. H. P. BASSETT (U.S.P. 1,562,490, 24.11.25. Appl., 19.2.23).—Arsenates are made from arsenious compounds and bases which form soluble chlorides and insoluble arsenates. The arsenite first formed is oxidised with chlorine in acid solution, and a further quantity of base is then added to make the solution alkaline and precipitate the arsenate. A. GEAKE.

Zirconium oxide complex. C. J. KINZIE, Assr. to TITANIUM ALLOY MANUF. CO. (U.S.P. 1,562,890, 24.11.25. Appl., 10.4.23).—Impurities are removed from zirconia by heating with a small quantity of a vitrifiable mixture. A. GEAKE.

Producing artificial cryolite, free from iron. H. SPECKETER, Assr. to CHEM. FABR. GRIESHEIM-ELEKTRON (U.S.P. 1,563,536, 1.12.25. Appl., 10.1.25).—Aluminium salt solutions containing iron are treated with not more than 9 mol. of hydrofluoric acid, and not less than 3 mol. of an alkali chloride per molecule of aluminium oxide; the precipitate thus obtained is treated with an alkali fluoride in the presence of water. A. GEAKE.

Separating soluble sodium salts. C. E. DOLBEAR (U.S.P. 1,563,613, 1.12.25. Appl., 17.12.23).—Sodium chloride, sulphate, and carbonate are separated by extracting the two last-named salts with sodium chloride solution. Sodium hydrogen carbonate is then precipitated by treatment with carbon dioxide, and sodium sulphate by the addition of ammonia. After distilling off the ammonia the liquor is used to repeat the cycle. A. GEAKE.

Separating constituents of mineral silicates. S. W. SCOFIELD and J. B. LA RUE (U.S.P. 1,563,875, 1.12.25. Appl., 4.2.24).—Amorphous potash felspar is completely converted into soluble substances by heating with 1—2 times its weight of 90% alkali hydroxide, together with its own weight of water, at 300 lb. pressure and 270°. Heating is continued until the normally insoluble constituents are fused, and more water is then added. A. GEAKE.

Absorption apparatus. (E.P. 236,918.)—See I.

Magnesia cement (E.P. 243,197).—See IX.

VIII.—GLASS; CERAMICS.

Fundamental law of annealing [glass]. F. W. PRESTON (Trans. Opt. Soc., 1924–5, 26, 270—273).—Assuming that annealing is a function of stress (f) and viscosity (η) only, dimensional analysis leads to the relationship $1/f-1/f_0=Bt/\eta$ (in which f_0 is the initial stress at $t=0$), a law identical with that obtained experimentally for glass by Adams and Williamson (cf. B., 1921, 81 A). If the modulus of rigidity (G) is included, the dimensional equation obtained is $-df/dt=f^2\phi\,(f/G)/\eta$. A. COUSEN.

Chrome aventurine glaze. S. KONDO (Tokyo Higher Tech. School, Bull. 3, 1925, 13—21).—In fritted glazes of the type (0·25 K_2O, 0·25 Na_2O, 0·5 RO) (0·05 Al_2O_3, 0·02—0·10 Cr_2O_3) (2·50 SiO_2, 0·50 B_2O_3), no crystals appeared when RO was BaO, CaO, or MgO, but a slight aventurine effect was obtained at cones 010a—1a when RO was PbO. In fritted glazes of the type (0·25 K_2O, 0·25 Na_2O, 0·50 PbO) (0·05 Al_2O_3, 0·02—0·10 Cr_2O_3) ($xSiO_2$, yB_2O_3), the best proportions for the production of the aventurine were x=1·65 and y=0·60, 0·02—0·04 Cr_2O_3 was completely or almost completely dissolved at cone 5a. On adding 0·02—0·10 equiv. of prepared chromic oxide crystals to frits of the type (0·25 K_2O, 0·25 Na_2O, 0·50 BaO or CaO or PbO) (0·05 Al_2O_3) (2·50 SiO_2, 0·50 B_2O_3), an aventurine effect was produced by the recrystallisation of the dissolved Cr_2O_3 in all cases when the glazes were cooled rather quickly. The best proportions of RO and Cr_2O_3 were 0·25 BaO and 0·25 CaO and 0·08—0·10 Cr_2O_3. Using frits of the composition (0·25 K_2O, 0·25 Na_2O, 0·25 BaO, 0·25 CaO) (0·05 Al_2O_3) (1·75—2·50 SiO_2, 0·50 B_2O_3), with added chromic oxide crystals, the best results was obtained with 0·10 Cr_2O_3; the glazes appear finer on chrome bodies than on white ones. Aventurine glazes with clean surfaces were obtained at cone 12 when 0·2 or 0·5 equiv. of chromic oxide crystals was added to frits of the type (0·25 K_2O, 0·25 Na_2O, 0·50 CaO) (0·05 Al_2O_3, 0·04—0·08 Cr_2O_3) (1·65 SiO_2, 0·30 B_2O_3) containing 0·08 or 0·05 equiv. of Cr_2O_3 respectively. With fritted glazes of the type (0·09 K_2O, 0·41—0·61 Na_2O, 0·50—0·30 CaO) (0·09 Cr_2O_3) (1·60—2·60 SiO_2, 0·50—0·00 B_2O_3), the aventurine appeared in glazes rich in B_2O_3 and alkalis. The best result was obtained with the glaze (0·09 K_2O, 0·41 Na_2O, 0·50 CaO) (0·09 Cr_2O_3) (1·80 SiO_2, 0·40B_2O_3) fired at cone 8. S. KONDO.

Chromate glaze. S. KONDO (Tokyo Higher Tech. School, Bull. 3, 1925, 23—26).—Glazes of the composition (0·90 PbO, 0·10 CaO) (0·20 Al_2O_3, 0·01—0·45 Cr_2O_3) (0·50 SiO_2) were fired at cone 05a—5a. On vitrified porcelain, the immature glazes had a red colour which showed a strong tendency to change to green as melting progressed. All the immature glazes showed red needles. Most of the mature glazes showed green or red crystals. No crystals appeared on porous biscuits. Glazes of the composition (0·90—0·50 PbO, 0·10—0·50 CaO) (0·05 Al_2O_3, 0·02—0·25 Cr_2O_3) (0·50—2·00 SiO_2) were fired at cones 05a—5a. No crystals appeared in glazes with PbO less than 0·70 equiv. Crystals can be obtained with glazes which contain less than 0·20 equiv. of Al_2O_3 and more than 0·5 equiv. of SiO_2. S. KONDO.

Analysis of refractory materials and other products rich in alumina. E. SCHÜRMANN and W. BÖHM (Chem.-Ztg., 1925, 49, 933—934, 958—959).—The separation of large amounts of aluminium as chloride by saturating the solution with hydrogen chloride (cf. Schürmann and Schob, B., 1924, 299) greatly facilitates the determination of the minor constituents of clays and other aluminous materials. 10 g. of the sample are heated with hydrofluoric and sulphuric acids to expel silica, and the solution is evaporated until the bulk of the sulphuric acid is removed. The residue is dissolved in 200 c.c. of hydrochloric acid, the solution cooled and saturated with hydrogen chloride, and the precipitate collected on glass wool and washed with concentrated hydrochloric acid. The filtrate is evaporated nearly to dryness, treated with hydrogen sulphide to remove any copper etc., oxidised with chlorine, and treated with ammonia to precipitate iron, titania, zirconia, and the remaining alumina. The precipitate is dissolved in hydrochloric acid, the iron separated by ether or by ammonium sulphide after addition of tartaric acid and ammonia, and the titanium and zirconium by means of cupferron. Nickel, manganese, and lime are determined in the ammoniacal filtrate as usual. Magnesia is precipitated with ammoniacal ammonium carbonate or, preferably, the solution is evaporated to dryness, and the residue heated gently to expel ammonium salts, and the residual magnesium and alkali sulphates are weighed. The sulphates are dissolved in water and converted into chlorides by treatment first with lead acetate, then, after removal of the lead sulphate, with hydrochloric acid. The excess of lead is separated with hydrogen sulphide, the solution evaporated to dryness, and the residue heated with thrice its weight of mercuric oxide until it ceases to evolve fumes. After extraction with hot water the insoluble magnesia is collected, ignited, and weighed. The filtrate is tested for potassium and lithium by any suitable method. A. R. POWELL.

Structure of α- and β-quartz. BRAGG and GIBBS.—See A., 1926, 13.

Electrolysis of soda-lime glass. MULLIGAN.—See A., 1926, 31.

Elastic after-effect of glass. KÖNIG.—See X.

PATENTS.

Manufacture of glazed or enamelled ware. I. TRAUBE (E.P. 221,830, 15.9.24. Conv., 14.9.23).—See G.P. 394,817; B., 1924, 749.

Producing enamels and glazes. F. SOMMER and M. GROTH, Assrs. to CHEM. WERKE VORM. AUERGES. (U.S.P. 1,564,907, 8.12.25. Appl., 17.5.23).—See E.P. 197,933; B., 1924, 382.

IX.—BUILDING MATERIALS.

Xylolith [from sawdust and magnesia cement]. R. GRÜN (Hauptversamml. Ver. Deuts. Steinholzfabr., 2.5.25. Reprint).—The setting of Sorel cement (magnesite cement) is generally ascribed to the formation of magnesium oxychloride; the author shows that the amount of magnesium chloride used is only a fraction of that required to form the oxychloride, whilst setting will also take place with salts other than chlorides or magnesium salts. A determination of the viscosity of suspensions of finely divided magnesium oxide in various setting agents and water shows that the setting of the cement is due to the

formation of magnesium hydroxide in a colloidal form, and not of magnesium oxychloride. Experiments carried out with magnesite obtained from various sources show how the strength, volume changes, and adhesion of the xylolith prepared from sawdust and magnesia cement vary with the grade of magnesite used. B. W. CLARKE.

Action of sodium and magnesium sulphates on calcium aluminates. G. R. SHELTON (Ind. Eng. Chem., 1925, 17, 1267—1270).—The hydration products of the calcium aluminates $3CaO,5Al_2O_3$, CaO,Al_2O_3, and $5CaO,3Al_2O_3$, of which the two last are important constituents of aluminous cement which resists well the disintegrating action of sulphate solutions, are hydrated tricalcium aluminate and amorphous matter, whereas with $3CaO,Al_2O_3$ hydrated tricalcium aluminate crystals alone appear. The amount of gel formed and the time required for complete hydration increase with the alumina content of the compound. Sodium sulphate solutions, and magnesium sulphate solutions below $0{\cdot}1M$, form large quantities of sulphoaluminate crystals, especially in dilute solution, amorphous grains in the original hydrated suspension being transformed in the process. Stronger magnesium sulphate solutions form gypsum crystals. With magnesium sulphate solution, crystalline aluminate grains become surrounded by layers of gel, containing magnesium hydroxide, protecting the grains from further action of sulphates. B. W. CLARKE.

Effect of size and shape of test specimen on compressive strength of concrete. H. F. GONNERMAN (Bull. 16, Structural Materials Research Lab., Lewis Inst., Chicago, Oct. 1925, pp. 16).—The 6 in. by 12 in. cylinder generally used for compression tests of concrete is satisfactory for aggregates up to 2 in. diameter. The ratio of diameter of cylinder to maximum size of aggregate should not be less than 3, with cylinders of length equal to two diameters. Cubes show a strength 13—18% higher than the corresponding cylinders. B. W. CLARKE.

Studies of bond between concrete and steel. D. A. ABRAMS (Bull. 17, Structural Materials Research Lab., Lewis Inst., Chicago, Oct., 1925. 20 pp.).—The bond resistance of a steel bar embedded in concrete depends on the quality of the concrete and the condition of the surface of the bar; factors which influence the compressive strength of the concrete, *e.g.*, water ratio, proportion of cement, and fineness of aggregate, will affect the bond in the same way. The maximum bond resistance is about 24% of the compressive strength of the concrete, with an end slip of 0·01 in., but slipping of the bar commences at about 55—60% of this load. The use of crude oil instead of mixing water or hydrated lime in place of cement reduces the bond resistance and compressive strength of the concrete. The allowance of 4% of the 28-day compressive strength of concrete as the bond strength for plain bars gives a factor of safety of $2\frac{1}{2}$—3. B. W. CLARKE.

PATENTS.

Magnesia cement. K. WERNER (E.P. 243,197, 24.1.25).—Silicic acid, supplied entirely in the form of a filling material containing at least 30% of silicic acid capable of combining with the surplus magnesium chloride solution, is added to magnesia cement to form a cheap cement for the production of castings required to keep a constant shape, *e.g.*, as a substitute for iron in making pattern plates. Residues obtained in the manufacture of aluminium sulphate and alum are suitable silicious materials. B. W. CLARKE.

Impregnation of wood. L. F. BLARINGHEM (F.P. 561,927, 8.2.23).—Green wood is impregnated with solutions of alkali dichromates containing alkali chlorides or fluorides and, if desired, colouring agents. In this way the proteins and reducing sugars of the wood cells are coagulated, the wood is hardened, and the time required for drying reduced by 50%, whilst deformation during drying is avoided. In order to obtain the wood in different shapes the green wood is first treated with the dichromate solution, then shaped, treated with the chloride or fluoride solution, and dried; wood so treated retains its shape indefinitely. A. R. POWELL.

Refractory cement with a base of zirconium ore. F. C. F. LE COULTRE, Assr. to SOC. D'ETUDE DES AGGLOMÉRÉS (U.S.P. 1,565,472, 15.12.25. Appl., 9.10.24).—See E.P. 224,214; B., 1925, 318.

Treatment of cementitious material. K. WINKLER (U.S.P. 1,565,839, 15.12.25. Appl., 14.12.20).—See E.P. 167,138; B., 1922, 503 A.

Bituminous emulsions [containing blue clay]. WAILES DOVE BITUMASTIC, LTD., and R. SHAW (E.P. 243,976, 5.2.25).

Making [bituminous] emulsions. L. KIRSCHBRAUN (E.P. 244,135, 10.6.24).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Thermal treatment of molten iron and its application to malleable cast iron. E. PIVOVARSKI (Stahl u. Eisen, 1925, 45, 2001—2004).—In agreement with the author's observations on the influence of thermal treatment on fluid iron (B., 1925, 805), experiments on malleable irons of different compositions showed that heating to 1400—1500° retarded decomposition of the carbide on subsequent annealing, the effect increasing with decreasing silicon content. Heating to a lower temperature (about 1300°) or to a higher temperature (above 1500°) had the opposite tendency. These effects persisted even after annealing for 60 hrs. An iron made by mixing two samples which had been heated to temperatures in the lower and higher temperature zones showed after annealing greater carbide decomposition than a similar iron which had been heated directly to approximately the same temperature (1450°)

in the intermediate zone. As the temperature to which the fluid iron was heated was raised the temper carbon subsequently deposited became finer but not to the same degree as in the experiments with grey iron. Annealing above 900° gave finer distribution of the temper carbon although the rate of carbide decomposition was not accelerated. Annealing at about 800° produced no refinement of temper carbon but increased the rate of graphite crystallisation. By combining these annealing treatments additive effects were produced. T. H. BURNHAM.

High-quality cast iron with high carbon and phosphorus contents as an electric furnace product. K. VON KERPELY (Stahl u. Eisen, 1925, **45**, 2004—2008).—High-quality cast iron containing less than 3·0% C and 0·3% P is usually made in the cupola from carefully calculated burdens, and casting involves certain technical difficulties. By treating cast iron in a Nathusius electric furnace of 4—6 tons capacity, equally good mechanical properties can be obtained when the carbon and phosphorus are above these limits, *e.g.*, with C 3·0—3·2%, P 0·2—0·8%. The quality is influenced by the special slag used, consisting of crude lime, fluorspar, and powdered coke, and the casting temperature (usually between 1500° and 1600°) has an important effect. Owing to the quick cooling of the iron the graphite and phosphide eutectic are uniformly distributed in a fine network. Analyses and mechanical properties of a large number of "heats" made when feeding the electric furnace by three cupolas having an output of 5000 kg. per hr. are tabulated. The cupolas were charged with low-quality scrap high in phosphorus, carbon, and silicon. Equally good tensile properties were obtained as when pig iron was used. The iron produced showed a fine fracture and was tough under the repeated impact test. It was resistant to high pressure and sudden changes of temperature. The ultimate strength increased as the cross-section of the test pieces decreased.
T. H. BURNHAM.

Influence of some elements on spheroidisation of carbides in steels. T. ISIHARA (Sci. Rep. Tôhoku, 1925, **14**, 377—390).—Cylindrical rods, prepared from a series of manganese, chromium, and copper steels, were annealed at 1100°, cooled to make the structure lamellar, and then heated at the rate of 2° per min. to the neighbourhood of the Ac1 transformation, and the degree of spheroidisation of carbides observed. The three series, each embracing 20 alloys, were prepared from Swedish iron and the appropriate alloying metal within the limits. C 0·51%, Mn 1·02% to C 2%, Mn 4·09%, C 0·45%, Cr 0·99% to C 1·85%, Cr 3·95%, and C 0·41% Cu 1·01% to C 1·98%, Cu 3·98%. The interval of temperature above the Ac1 point within which spheroidising occurs is increased slightly with the content of manganese, and considerably with chromium, whilst copper has an intermediate effect. If the content of these elements in steels be kept constant the interval remains nearly constant up to the eutectoid concentration and then increases rapidly with increase in the content of carbon.
C. A. KING.

Structural diagrams of some special steels. T. KASÉ (Sci. Rep. Tôhoku, 1925, **14**, 453—478).—The structural diagrams of nickel, manganese, and chromium steels, made from Swedish iron, sugar charcoal, and the alloying element, were examined with reference to hardness (scleroscope), microstructure, and transformations, especially at low temperatures. Alloys which have completed the Ar3 transformation at atmospheric temperature do not undergo any marked change in physical properties when cooled in liquid air, but those having an austenitic structure may be transformed into martensite and expand abnormally, with increase in hardness. Thus an increasing content of nickel in nickel–iron alloys causes a rapid depression of the Ar3 point, which in the case of a 26% Ni alloy falls below 20° and in that of a 35% Ni alloy below −190°. Annealed nickel–iron alloys containing 20—34% Ni increase in hardness after being immersed in liquid air. The alloys, *e.g.*, iron–carbon–nickel (up to 10% Ni), which exhibit stepped transformations in magnetic analysis, assume a martensitic structure on cooling in virtue of the lower transformation temperature (below 300°). The structural diagrams of the steels examined are different from those described by Guillet, in that when the former are cooled slowly from the melted condition, the structural fields, after an addition of a small quantity of carbon, are bordered by straight lines almost parallel to the axis of the carbon. C. A. KING.

Influence of the thermal zone of working on the selection of steels for the valves of aeroplane engines. C. GRARD (Compt. rend., 1925, **181**, 1143—1145).—The most suitable alloy examined was a steel containing C 0·4, Si 2·5, Cr 12%, quenched in air at 1200° and reheated to 900°. This steel possessed the following characteristics: Ac 950°, *d* 7·8, tensile strength 92, elongation 12%, Brinell hardness 280 at ordinary temperature, nearly 40 at 900° (this latter value may be slightly increased by addition of 1·5% of tungsten), resistance to repeated impact 18,140. Valves made of this alloy retained their original polish and texture after continuous operation for 50 hrs. S. I. LEVY.

Quenching of light aluminium–copper alloys containing more than 5% of copper. L. GUILLET and J. GALIBOURG (Compt. rend., 1925, **181**, 1107—1108).—The hardness and electrical resistivity of castings containing 7—45% Cu, after quenching and subsequent reheating, are tabulated. Quenching and reheating greatly increase the hardness, which is more than doubled by the optimum treatment in some cases. The resistivity is abnormal, being increased not only by quenching, but also in some cases by the subsequent annealing. Both properties increase steadily with increasing copper content.
S. I. LEVY.

Effect of cold working and annealing on some physical properties of copper, aluminium, and their alloys. T. MATSUDA (Sci. Rep. Tôhoku, 1925, 14, 343—376).—The physical properties of copper, brasses (Cu 71, 60, and 54%), aluminium-bronze (7% Al), phosphor-bronzes (Sn 3 and 9%), manganese-copper (Mn 3·6%), aluminium, and duralumin, were determined after cold rolling and annealing. The electrical resistance increased as the result of cold deformation whether the deformation was produced by rolling, drawing, or twisting, but there was no systematic differences in the resistance of cold-drawn plates measured along or transversely to the direction of rolling. The increase in resistance is considered to be due to straining of the space lattice and not to the formation of fine cracks as suggested by Tammann. Lower strength and higher elongation were observed in specimens cut longitudinally to the direction of rolling. When the alloys were heated the strain in the space lattice began to disappear at temperatures much below that at which recrystallisation commenced, and the first step in the decrease of electrical resistance corresponded with this release of strain, the second change in resistance occurring during the recrystallisation. On heating, the alloys expanded in the direction of rolling and contracted in the transverse direction, but the internal strain produced by cold rolling is considered not to exert much influence on the electrical resistance.

C. A. KING.

Determination of certain curves [of the lead–zinc–silver system] and their application to the desilverisation of lead by the Parkes process. G. K. WILLIAMS (Proc. Austral. Inst. Min. Met., 1925, [58], 47—97).—As a result of a detailed investigation of the behaviour of ternary alloys at the lead corner of the lead–zinc–silver system a diagram has been constructed giving the cooling curves, temperature contours, and eutectic compositions of this system so that the behaviour of any lead–silver alloy containing less than 100 oz. of silver per ton may be accurately followed during desilverisation by the Parkes process. The composition of the lead bullion obtained after the zinc treatment is dependent on the amount of silver present and on the amount of zinc added, but the final alloy is always a eutectic having a composition corresponding with some point on the line joining the lead–silver eutectic point to the lead–zinc eutectic point. The crust that separates from the molten ternary alloys is essentially a solid solution of lead, silver, and zinc, and may contain 5—10% Pb other than that entangled in the crystals. The statement that a minimum amount of 0·7% Zn must be present in the lead before any desilverisation will take place is erroneous, the amount of zinc necessary depending entirely on the silver content of the lead before desilverising; the higher the silver up to 2·5% the lower will be the quantity of zinc required to remove it. After desilverisation the lead usually contains about 0·6% Zn and 0·2 oz. Ag per ton as the Pb–Zn–Ag eutectic. In applying these facts to practice considerable variation in the results and working costs may be obtained by varying the mode of application of the zinc, temperature of stirring, temperature of skimming, and proportion of zinc added. When only one zinc treatment is used, effective desilverisation requires a larger amount of zinc, and a correspondingly large weight of crust is formed if the skimming is done in one operation at a temperature just above the eutectic temperature. If, however, the crusts that form first are removed at a comparatively high temperature and set aside for recovery of the silver, and the later crusts are removed at the eutectic temperature and returned to the next charge, then the zinc consumption is lower, the first crusts are richer in silver, and the time of desilverisation is shortened; on the other hand, the fuel consumption is very high, the strain on the workman is greater, and the weight of crusts for re-treatment is comparatively large. At the Port Pirie smelter two zinc treatments are given; lead containing 60 oz. Ag per ton is heated to 450° and crusts from the second zinc treatment of a previous charge are added. After cooling to the temperature at which the lead begins to solidify, the crusts are removed with a Howard press and set aside for the recovery of the silver. The metal is again heated to 450°, new zinc added to bring the zinc content to about 0·7%, and after cooling as before, the crusts are removed and used for the first treatment of the next batch. The final crusts are heated to 650—700° and allowed to cool until the zinc layer solidifies; this contains 16,000 oz. Ag per ton and is sent direct to the retorts. The lead layer is allowed to cool to 350° and the crust that separates is added to the next lot of crust to be enriched, whilst the remaining lead is returned to a main charge before the second zinc treatment. Tables showing the requisite quantities of zinc required for the first and second treatments of lead containing varying amounts of silver are given.

A. R. POWELL.

System lead–antimony. II. R. S. DEAN, W. E. HUDSON, and M. F. FOGLER (Ind. Eng. Chem., 1925, 17, 1246—1247; cf. B., 1923, 836 A; 1924, 835).—The extent to which antimony forms solid solutions with lead has been determined at the eutectic temperature (247°) by thermal analysis, and over a range of temperatures from 20° to 238° by observing the break in the composition–electrical conductivity curves. The solubility is 2·45% Sb at 247°, and falls to 0·80% at 20°. Lead–antimony alloys containing antimony in excess of the amount soluble at room temperature, when quenched from higher temperatures, subsequently age-harden at room temperature, as indicated by increase in tensile strength. The amount of hardening shows a maximum near 2·5% Sb. The increase in conductivity during the hardening process indicates that the solid solution formed at higher temperatures is retained by quenching and subsequently breaks down by separation of a constituent in a fine state of subdivision.

A. B. MANNING.

Elastic after-effect [of metals and glass] at different temperatures. H. KÖNIG (Physikal. Z., 1925, 26, 797—811).—Determinations have been

made by Bennewitz's method of the rate of bending and recovery of thin rods of glass, copper, brass, and aluminium subjected to fixed loads at temperatures between 15° and 360°. The loading and recovery curves were approximately logarithmic in nature, but in the majority of cases the deformation of the rods increased rather more quickly than the logarithm of the duration of the load, although for glass between 150° and 250° the increase was slower. The logarithmic law is approximately applicable for times up to about 3000 sec. for certain temperatures, but, to within experimental error, it is valid only up to 100 sec. The loading curves may be approximately reproduced by the formula $y=y_0+at^n$, where a and n are constants. The recovery curves were usually represented approximately by the application of the superposition principle to the loading curves. The calculated recovery curves were usually somewhat flatter than the experimental ones, the discrepancy being greater for glass than for the metals. The flow of the metals at high temperatures enhanced this discrepancy, but when this effect was neglected the after-effect was found to be roughly proportional to the loading. The variation of the after-effect with temperature was greater with the metals than with glass. It was especially large in the case of brass, the deformation of which in a given time interval was 100 times as great at 300° as at 16°. The rate of deformation in the case of metals increases with rise of temperature. With glass, a notable decrease of deformability occurs in some temperature ranges, although in others a marked increase occurs. The after-effect is greatly dependent on the pre-treatment of the specimen. In the case of glass preheating generally decreases the after-effect. The after-effects in copper showed a complex behaviour under different conditions of preheating, due apparently to recrystallisation changes during the heat treatment.

F. G. TRYHORN.

Gases in metals. II. Determination of oxygen and hydrogen in metals by fusion in vacuum. L. JORDAN and J. R. ECKMAN (Sci. Papers U.S. Bur. Standards, 1925, **20**, [514], 445—482).—Twenty to 60 g. of metal are fused in a gas-free graphite crucible in a vacuum in a high-frequency induction furnace. A crucible of electrically fused magnesia around the crucible serves as a radiation screen and heat insulator. The double crucible is set inside a silica tube within the furnace and the arrangement permits of maintaining the molten metal at 1500° while the furnace walls remain comparatively cool. Water vapour and carbon dioxide are determined directly and carbon monoxide and hydrogen indirectly by passing the gases evolved from the heated sample through a train of solid absorbents at low pressures. Phosphorus pentoxide and ascarite (a granular sodium hydroxide–asbestos absorbent) are used for the absorption of water and carbon dioxide respectively, and an electrically heated copper oxide furnace for the oxidation of carbon monoxide and hydrogen forms part of the train. The method does not distinguish between oxygen present in the metal as carbon monoxide or dioxide or oxides of non-metallic inclusions or of metallic constituents. The recovery of oxygen is complete from oxides of iron, manganese, silicon, aluminium, titanium, and zirconium, and the determination of oxygen in iron and steel is not interfered with by the presence of sulphur. The method is suitable for the determination of oxygen in iron, steel, cast iron, and many non-ferrous metals and alloys. Direct fusion of low-carbon steels in refractory-oxide crucibles or the Goerens–Paquet method (fusion of the sample with an antimony–tin alloy in similar crucibles) does not determine all the oxygen present in the metal. The reduction of the refractory oxides by the carbon in the steel is an additional difficulty which becomes more pronounced with higher carbon content. The use of the graphite crucible permits of a satisfactory determinationte oxygen irrespective of the amount of carbon in the steel.

M. COOK.

Copper from its sulphide ores. R. SAXON (Chem. Trade J., 1925, **77**, 626—627).—Iron pyrites gives a solution of ferrous and ferric sulphates when electrolysis takes place in a medium of 10% sulphuric acid, using an iron cathode surrounded by the mineral and a carbon anode. The proportion of ferric sulphate in solution increases with time and eventually ferric hydroxide is precipitated. Copper pyrites with similar treatment gives a solution of ferrous sulphate, copper sulphide being deposited on the cathode. If the electrolyte and cathode be then removed and the latter be used as an anode, copper can be deposited on a new ferrochrome cathode. An apparatus for effecting this without changing the electrolyte is described.

L. M. CLARK.

Extraction of copper from burnt pyrites by means of sulphuric acid. N. F. JUSCHKEVITSCH (Russian J. Chem. Ind., 1924, **1**, [1], 5—11).—In the laboratory 86% of the total copper may be extracted from burnt pyrites by boiling dilute sulphuric acid, but on a works scale the extraction is less complete owing to the slowness with which the copper is dissolved. The amount of acid required is considerable even when the percentage extraction is low and increases to 11·3 kg. of monohydrate per kg. of copper, when 85·5—86% of the latter is extracted. Increase in the concentration of the acid effects neither improved extraction of the copper nor diminution of the amount of acid required. This method seems too costly for practical application. Better results are obtained by subjecting the burnt pyrites to preliminary sulphatisation by heating at 500—600° and afterwards treating with water and sulphuric acid solution, as much as 97% of the total copper being readily extracted in this way with an expenditure of 8 kg. of chamber acid per 100 kg. of pyrites. The sulphatisation is expensive as regards both fuel and labour if carried out in separate furnaces, but it could be cheapened by using a mechanical furnace and by either atomising the sulphuric acid or introducing sulphur dioxide into the lower zones of the furnace.

T. H. POPE.

Zinc compounds at high temperatures. [Direct production of volatilised zinc oxide from sulphide ores.] W. G. WARING (Min. and Met., 1925, 6, 610—613).—Zinc sulphide may be volatilised directly from complex ores containing zinc blende, galena, pyrites, etc., and burnt directly to pure white zinc oxide which may be selectively condensed. The high temperature produced by the combustion of zinc sulphide in a confined space is sufficient to keep the oxide formed in the state of vapour, which condenses at 1370° leaving the more volatile impurities (basic lead sulphate, bismuth, antimony, arsenic, cadmium, selenium, and tellurium oxides) which condense at temperatures below 1060°, to be collected in a second receiver. Experiments carried out in a combined reverberatory-blast furnace (U.S.P. 1,513,775; B., 1925, 14), using a shallow charge and oxidising the volatilised sulphides at the surface by means of a hot blast, resulted in the production of a zinc oxide product containing 97% ZnO with a loss of only 2% of the total zinc in the slag and flue dust; at the same time a high-grade copper matte containing the gold and most of the silver was obtained. The zinc oxide was purified by digesting it under slightly reduced pressure in iron vessels with 4*N*-ammonium carbonate at the ordinary temperature. In 1 hr. all the zinc dissolved together with the cadmium and a small quantity of silver, leaving a residue of basic lead carbonate and other impurities. After settling and filtering, the solution was passed through a cylinder containing scrap zinc to remove cadmium and silver and then distilled with live steam, whereby the zinc was deposited as a light, non-adherent powder of zinc carbonate which could readily be converted into oxide by heating at 400°. The ammonia and carbon dioxide were recovered for use again. A. R. POWELL.

Reduction of zinc oxide by carbon. G. A. ZELLER and B. M. O'HARRA (Sch. Mines Met. Univ. Tech. Bull., 8, 3—32; Chem. Abstr., 1925, 19, 3451). —At temperatures above that at which the reduction of zinc oxide by carbon in a sealed graphite retort began, the rate of reduction was doubled with equal intervals of temperature, the intervals varying for different forms of carbon. Calcined zinc carbonate was reduced much more rapidly than other forms of zinc oxide. The differences are due mainly to differences in the condition of the surfaces.
A. A. ELDRIDGE.

Determination of silver in zinc retort residues. V. HASSREIDTER (Metall u. Erz, 1925, 22, 403—404; Chem. Zentr., 1925, II., 2219).—Zinc retort residues usually have such a high carbon content that an oxidising charge is necessary in order to get a satisfactory lead button for the determination of silver. The following charge is recommended: 15 g. of residues, 75 g. of lead dioxide, 10 g. of sand, and 45 g. of sodium carbonate. If the button weighs more than 60 g. the requisite amount of nitre should be added in order to obtain a 60-g. button, which is then scorified and cupelled as usual.
A. R. POWELL.

Method of reducing the corrosion of water conduits consisting of pipes of two different metals. H. CASSEL (Korrosion u. Metallschutz, 1925, 1, 75; Chem. Zentr., 1925, 96, II., 2024).—Considerable corrosion takes place at copper–iron joints in water conduits. This may be greatly reduced by inserting a short length of lead piping between the copper and the iron pipes. The lead piping becomes coated with a thin layer of lead peroxide and acts as an inert body and, owing to the separation of the iron and copper and to the poor conductivity of ordinary water, no current flows between the two metals and hence corrosion is slight. A. R. POWELL.

Natural water corrosion and hydrogen-ion concentration. J. R. BAYLIS (Chem. Met. Eng., 1925, 32, 974—975).—Experiments confirm the electrochemical theory of corrosion. In no case was a concentration of 3 p.p.m. of soluble iron found in a solution free from oxygen with iron present when the p_H was between 8 and 9. Soluble iron in water with excess of dissolved oxygen oxidises at a much faster rate at p_H 9·0 than at p_H 6·0. If an alkalinity of 50 p.p.m. be assumed, then at p_H 8·0 less than 0·1 p.p.m. of soluble iron is found in the absence of oxygen, about 0·1 at p_H 8·0, about 1·0 at p_H 7·5, and about 4·0 at p_H 7·0. At less than p_H 7·0 the soluble iron increases rapidly. "Tuberculous" deposits form an impervious coating where soluble iron from the interior comes into contact with dissolved oxygen. If the water has p_H 8·5 the iron forms an impervious coating of crystalline iron oxide in a few months. Pits are often found under tubercles and negative ions, such as sulphate and chloride ions, are concentrated in these: the overlying tubercles may contain over 1% of these salts. The volubility of ferrous carbonate determines the likelihood of the water to stain. H. MOORE.

Mechanism of plastic deformation [of metals]. H. MEYER and F. NEHL (Stahl u. Eisen, 1925, 45, 1961—1972).—A review of the literature of the subject is given and special attention is devoted to considering the "slip-cone" theory of deformation. The formation of cones on compressing cylindrical test-pieces of steel is ascribed to frictional forces between the jaws of the compressing machine and the end surfaces of the test-piece: the lines of stress in the test-piece are brought out by etching. By using the fact that the yield load in compression depends on the height of the test-piece when end surface frictional forces are not eliminated, a method is devised whereby the angle of inclination of the plane of deformation proceeding from the end surface can be measured for iron at the yield point. By measuring the amount of crushing in cylindrical test-pieces undergoing more severe deformation, it was found that to produce a given decrease in height always required much heavier loading than would be expected by calculation: this is also explained by the influence of frictional forces at the end faces. The factors bearing on the work needed to cause plastic deformation are discussed, and the

mechanisms of slight and severe deformation in a test-piece with non-rectilinear cross-section, are explained on the basis of experiments with double cones of mild steel. Information on the rolling of iron is obtained by etching cold-rolled square bars, when lines of stress are revealed. L. M. CLARK.

Froth flotation explained by *X*-rays. C. G. MCLACHLAN (Eng. Min. J. Press, 1925, 120, 408—409; Chem. Abstr., 1925, 19, 3450).—Space-lattice diagrams show that in non-floatable minerals oxygen is present at the surface, with consequent small attraction for air bubbles, whilst the surface of floatable minerals contains metal or sulphur.
A. A. ELDRIDGE.

See also A., 1926, 17, **Remanent magnetism and specific resistance of pure iron–carbon alloys** (CAMPBELL). 20, **Production of oxide films on copper at ordinary temperature** (EVANS). 30, **Electromotive behaviour of aluminium** (DE GRUYTER). 33, **Effect of differential aeration on corrosion** (MCAULAY and BOWDEN). 41, **Volumetric determination of iron in hydrochloric acid solution** (MANCHOT and OBERHAUSER); **Separating cadmium, bismuth, and lead** (MANCHOT, GRASSL, and SCHNEEBERGER).

PATENTS.

Non-magnetic steel wire. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC AND MANUFACTURING Co. (U.S.P. 1,561,306, 10.11.25. Appl., 21.4.21).—A non-magnetic ferrous alloy contains 10—18% Mn, 2—8% Cr, and 5—10% Ni. T. H. BURNHAM.

Tool [steel]. E. G. HONES (U.S.P. 1,561,857, 17.11.25. Appl., 29.6.25).—Tools are made of alloy steel containing 0·1—3·0% Cu, 0·1—3·0% Zr, and 0·1—3·0% Co. T. H. BURNHAM.

Heat-resisting steel. E. H. NOACK (U.S.P. 1,562,782, 24.11.25. Appl., 14.6.24).—The steel contains Si 1·50—2·00%, Cu 0·50—1·00%, Mn not more than 0·12%, and C not exceeding 0·10%.
M. COOK.

Heat-resisting [iron] alloy. N. V. HYBINETTE (U.S.P. 1,563,573, 1.12.25. Appl., 11.6.23).—Heat-resisting alloys contain Cr 10—20%, Ni 30—40%, C 0·4—1·5%, less than 1% Si, and the remainder iron. C. A. KING.

Manufacture of alloy steels and iron. B. D. SAKLATWALLA (U.S.P. 1,564,139, 1.12.25. Appl., 8.4.24).—An alloying metal is introduced into iron or steel by electrolysing a molten bath containing an unreduced compound of the alloying metal and allowing the metal to descend into a bath of molten iron beneath the electrolyte bath. C. A. KING.

Process of making ferrovanadium. L. F. VOGT, Assr. to STANDARD CHEMICAL Co. (U.S.P. 1,564,156, 1.12.25. Appl., 26.12.24).—A mixture of a finely-divided fluxing agent containing lime and a reducing agent containing silicon is spread over a slag layer which contains vanadium compounds and which covers a molten bath of metal. The mixture of reducing agent and flux fuses and descends gradually through the slag, reducing the vanadium compounds during its progress. C. A. KING.

Carburising process. N. E. PHILLIPS (U.S.P. 1,561,482, 17.11.25. Appl., 27.8.23).—The iron is enclosed in a thick, closely consolidated packing of pulverised carboniferous material, which is itself enclosed in a similar packing of a powdered material containing calcium. The temperature is raised and the iron maintained at a white heat for 4—10 hrs., according to the depth of carburisation desired. T. H. BURNHAM.

Cold processes for drawing and rolling metals. J. G. DE LATTRE and H. HARDY (E.P. 226,774, 20.6.24. Conv., 29.12.23).—Before rolling or drawing, the metal is coated by dipping into a molten alloy of a soft metal, such as lead, with one or more metals, such as copper, nickel, or tin, present in sufficient quantity to form a large number of small ducts in the covering layer after the soft metal has been removed in rolling or drawing. The preferred composition of the covering alloy is Pb 60, Ni 2, Sn 10, and Cu 28 pts. M. COOK.

Electrolytic separation of metallic chromium. Production of solutions containing oxides of chromium for the deposition of metallic chromium. E. LIEBREICH (E.P. [A] 237,288, 20.7.25, and [B] 243,046, 13.8.24. [A] Conv., 21.7.24). —(A) A solution for the electrolytic deposition of chromium is prepared by the electrolytic reduction of a solution in water of chromium trioxide containing less than 1·2% of free sulphuric acid. A low current density is used so that no metal is deposited until a sufficient quantity of lower oxide has been formed. (B) Chromium trioxide containing less than 1·2% of free sulphuric acid is heated at 170—200° in the absence of air and without stirring until a slightly pasty mass is obtained. This is dissolved in water and the acidity adjusted to 0·6—0·8% of free sulphuric acid for matte deposits and to 0·8—1·2% of free acid for bright deposits of metallic chromium.
A. R. POWELL.

Leaching processes for extraction of metals from ores and the like. W. G. PERKINS, and METALS PRODUCTION, LTD. (E.P. 243,075, 22.8.24 and 5.3.25).—After treatment of the ore by ammoniacal solutions a mixture of steam and ammonia, with or without carbon dioxide, is applied. The presence of ammonia, assisted if necessary by carbon dioxide, prevents the re-precipitation of copper. The vapours may be introduced above the ore while this is in contact with leaching liquor. The liquid condensed in the charge from the vapours is collected separately from the leaching liquors and used to make up fresh leaching solution. Copper ores rendered porous by heating (cf. E.P. 185,242 and 190,246; J., 1922, 863 A; 1923, 147 A) are treated first with ammoniacal carbonate solutions and then with the mixture of steam and ammonia with or without carbon dioxide. The process is

primarily for the treatment of oxidised copper ores but is applicable to all cases where ammoniacal leaching solutions are used. M. Cook.

Condensing zinc vapour. O. Ravner, Assr. to Det Norske Aktieselskab for Elektrokem. Ind. (U.S.P. 1,562,473, 24.11.25. Appl., 7.3.21).—The condenser comprises a revolving drum connected with a stationary tube in such a way that the condensate formed in the tube falls back automatically into the drum. M. Cook.

Deoxidising metals and alloys. A. Pacz (U.S.P. 1,562,655, 24.11.25. Appl., 11.5.21).—An intimate mixture of aluminium powder and sodium silicofluoride is added to the molten metal or alloy. M. Cook.

Treating metallic objects. Coated metallic article. C. P. Madsen, Assr. to Madsenell Corp. (U.S.P. 1,562,710—1, 24.11.25. Appl., 27.7.20. Renewed 10.2.25).—(a) An iron or steel surface is prepared for the application of an adherent impervious metallic coating by arranging it as anode in an electrolytic bath containing sulphuric acid of a strength of 86% or over, maintained at a temperature above normal. (b) A flexible metallic base is provided with an adherent workable coating of a "nickel metal" deposited electrolytically. H. Holmes.

Treating manganese-silver ores. M. F. Coolbaugh and J. B. Read, Assrs. to Complex Ores Recoveries Co. (U.S.P. 1,562,942, 24.11.25. Appl., 4.3.22).—Ores containing silver, manganese, and sulphides are roasted under such conditions that the silver sulphide is not affected whereas other sulphides are oxidised. The ore and evolved gases then pass together to a cooler zone where the manganese is converted into sulphate, which may be separated from the silver sulphide by extracting with water. A. Geake.

Roasting ores. A. D. H. L. Fassotte, Assr. to Comp. des Métaux Overpelt-Lommel (U.S.P. 1,563,616, 1.12.25. Appl., 16.1.24).—A gaseous current with ore particles in suspension travels up a central column and down a surrounding annular column where the heat developed is utilised to preheat the ore particles, most of the necessary air being preheated by heat from the waste gases. The ore together with a small quantity of preheated air is fed into the base of the central column horizontally and the main portion of the preheated air meets the mixture at right-angles. The velocity of the upward gaseous current is gradually reduced and the remainder of the air, which is not preheated, is injected into the central column at different heights. M. Cook.

Precipitating gold from solutions. W. H. Bates (U.S.P. 1,563,957, 1.12.25. Appl., 2.7.23).—In the electrolysis of solutions containing precious metals a mercury cathode having a large active area is used in close proximity to an anode also of large active area. C. A. King.

Open-hearth furnace. C. Davies, jun. (U.S.P. 1,564,049, 6.12.25. Appl., 6.4.22).—An air-port extends across the end of the hearth of a furnace and a fuel-supply duct is provided to deliver fuel in a direction substantially parallel to the air-port and beneath the issuing stream of air. The fuel-supply duct is of approximately the same transverse width as the air-port. D. F. Twiss.

Removal of gases from molten aluminium. T. Staub (F.P. 591,145, 6.12.24).—Aluminium is melted under a slag of dehydrated sodium and calcium chlorides, potassium sulphate, and cryolite to remove gases and slag-forming impurities. A. R. Powell.

Gray cast iron. K. Sipp, Assr. to H. Lanz (U.S.P. 1,564,284, 8.12.25. Appl., 15.5.24).—See E.P. 225,501; B., 1925, 596.

Detinning of tinned-iron scrap. H. Davies and M. A. Adam (U.S.P. 1,565,425, 15.12.25. Appl., 28.6.23).—See E.P. 209,124; B., 1924, 180.

Melting and refining non-ferrous metals. Barrett Co., Assees. of D. Cushing (E.P. 225,871, 6.12.24. Conv., 7.12.23).—See U.S.P. 1,556,591; B., 1925, 996.

Manufacture of alloys. H. Wade. From Internat. Nickel Co. (E.P. 243,892, 21.11.24).—See U.S.P. 1,533,818; B., 1925, 510.

Treatment of ores or residues containing zinc. S. C. Smith (U.S.P. 1,565,215, 8.12.25. Appl., 14.5.24).—See E.P. 237,306; B., 1925, 767.

Producing zinc or zinc and lead. C. E. Cornelius, Assr. to Trollhättans Elektrothermiska Aktiebolag (U.S.P. 1,566,183, 15.12.25. Appl., 27.7.21).—See E.P. 171,722; B., 1922, 62 a.

Muffles for the heat treatment of metals. J. H. Lloyd, C. M. Walter, and J. Palser (E.P. 244,280, 3.1.25).

Nickel catalyst (U.S.P. 1,563,587).—See I.

XI.—ELECTROTECHNICS.

Amount and distribution of light emitted by carbons of high luminous intensity used in projection. J. Rey (Compt. rend., 1925, **181**, 1133—1134).—Photometric examination of the image of the crater of an arc formed between carbon electrodes having a core of rare-earth salts, shows the existence of several distinct zones, the average amount of light for the whole area of the crater being 296·3 candles per sq. mm. with a current density of 0·75 ampère per sq. mm. The figures obtained are much below those claimed by the makers. S. I. Levy.

Sulphuric acid from a quadruple mixture by electrolysis. Saxon.—See A., 1926, 34.

Electric vacuum furnace. Partington and Anfilogoff.—See A., 1926, 41.

Photo-electric radiation pyrometer. LINDEMANN and KEELEY.—See I.

Obtaining iodine by electrolysis. PISARSHEVSKI and TELNI.—See VII.

PATENTS.

Resistance material electrically conductive in a cold state and usable at high temperatures. T. A. F. HOLMGREN (E.P. 228,142, 12.1.25. Conv., 22.1.24).—Silicon carbide or other similar carbide, *e.g.*, titanium carbide, is mixed with a refractory binding medium consisting wholly or partly of an oxide of a metal of the alkaline-earth or magnesium groups, and the mixture burnt at a temperature not lower than about 1200° in an atmosphere containing carbon monoxide. If desired, one or more refractory metals or graphite may be incorporated in the mixture, and the surface of the conductor may be coated with a glaze consisting, *e.g.*, of 0·1 pt. of barium oxide, 0·2 of calcium oxide, 0·2 of potassium oxide, 0·1 of magnesium oxide, 1·0 of alumina, and 7·0 pts. of silica. J. S. G. THOMAS.

Synthesising gases in electric furnaces. M. J. BROWN, Assr. to ROESSLER AND HASSLACHER CHEMICAL Co. (U.S.P. 1,562,684, 24.11.25. Appl., 2.10.22).—In an electric resistance furnace for effecting gaseous reactions, and having a resistor of finely-divided material arranged between the electrodes, the resistor material is supplied at such a rate with the reacting gases that the value of the electrical resistance is maintained within predetermined limits. J. S. G. THOMAS.

[Evacuating] electron discharge devices. WESTERN ELECTRIC CO. From WESTERN ELECTRIC CO., INC. (E.P. 243,200, 31.1.25).—Residual gas is removed from an electron discharge device by means of a getter, *e.g.*, of aluminium, mounted in such a way that the vapour emitted by the getter, which is vaporised by an alternating current, does not attack the electrode surfaces. J. S. G. THOMAS.

Means for automatically interrupting the current in electric furnaces if a fault occurs in an electrode. RHEINISCHE METALLWAAREN- U. MASCHINENFABRIK (E.P. 241,865, 24.8.25. Conv., 22.10.24).

Dehydrating oil emulsions (U.S.P. 1,562,712).—See II.

Silicic acid (U.S.P. 1,562,940).—See VII.

Incandescence bodies for electric lamps (E.P. 243,251).—See VII.

XII.—FATS; OILS; WAXES.

Oils of chaulmoogra group. E. ANDRÉ (Compt. rend., 1925, **181**, 1089—1091).—The oils and fats of the chaulmoogra group used in the treatment of leprosy are all strongly dextrorotatory and have closely similar physical properties. Fractionation with organic solvents indicates that the high dextrorotation is not due entirely to the content of chaulmoogric and hydnocarpic acids and it is probable that strongly dextrorotatory liquid fatty acids are present. The origins and physical properties of the oils studied are as follow:—*Taraktogenos Kurzii* (chaulmoogra) oil: d^{32} 0·9425; α_D (5% chloroform solution), +48·0°, m.p. 33—39°; saponif. value, 210·4; iodine value (Hanus), 96·1. *Hydnocarpus anthelmintica* (kraboa) oil (two samples): d^{29} 0·9447, d^{32} 0·9427, n_D^{29} 1·4755, 1·4742, α_D +58·17°, 48·0°, m.p. 26—29°, 25—26°, saponif. value 191, 187·3, iodine value 90, 88·3. *Hydnocarpus Wightiana* (kavatel) oil: d^{32} 0·9330, n_D^{29} 1·4780, α_D +61·67°, m.p. 28—32°, saponif. value 197·2, iodine value 103. *Hydnocarpus alpina*: d^{32} 0·9346, n_D^{32} 1·4764, α_D +57·0°, m.p. 20·5°, saponif. value 201, iodine value 95. *Asteriastigma macrocarpa*: d^{32} 0·9217, n_D^{25} 1·4725, $[\alpha]_D$ +44·0°, m.p. 37—39°, saponif. value 189·4, iodine value 82·8. *Oncoba echinata* (gorli) fat; d^{32} 0·9286, n_D^{31} 1·4740, $[\alpha]_D$ +56·17°, m.p. 40·5—41·5°, saponif. value 184·5, iodine value 98. *Carpotroche brasiliensis*: d^{32} 0·9499, n_D^{31} 1·4755, α_D+53·67°, m.p. 21—23°, saponif. value 183·7, iodine value 106·1. The "chaulmoogra oils" of commerce appear to be of various botanical origins. L. F. HEWITT.

Higher fatty acids of peanut [arachis] oil. W. D. COHEN (Proc. K. Akad. Wetensch. Amsterdam, 1925, **28**, 630—635).—Repeated fractional distillation in a vacuum (0·001 mm.) of the ethyl esters of the higher fatty acids present in arachis oil effects a separation into two fractions, (1) m.p. 42·5°, (2) m.p. 53·5—54°, which on hydrolysis yield respectively arachidic acid, m.p. 74·5—75°, and a C_{24} acid, m.p. 80—80·5°, the properties of which render it probable that it is a straight-chain acid belonging to the same homologous series; each acid is present to the extent of 3—4% of the total fatty acids. No trace of a C_{22} acid could could be detected, the substance obtained by Ehrenstein and Stuewer (B., 1923, 1031 A) being probably a mixture, since effective separation cannot be obtained by distillation in an ordinary filter-pump vacuum. J. W. BAKER.

Detection of unsaponifiable matter in marine animal oils. D. HOLDE and A. GORGAS (Chem. Umschau, 1925, **32**, 314—316).—The qualitative Spitz-Honig test for unsaponifiable matter was found to fail with two marine animal oils known to contain 6% and 9% of unsaponifiable matter respectively. The failure of the test, which depends on the formation of a turbidity on adding water to the oil saponified by alcoholic alkali, was traced to a surface tension effect, whereby the unsaponifiable matter remained floating on the surface as droplets instead of forming a cloudy emulsion. This anomalous behaviour is determined by the nature of the unsaponifiable matter and the soap present. The test is made more trustworthy in the case of certain marine animal oils containing highly unsaturated hydrocarbons if the saponified alcoholic solution of the oil be diluted with water added drop by drop from a burette. A. DE WAELE.

Application of measurement of capillary forces to study of fatty acids. R. Dubrisay (Compt. rend., 1925, **181**, 1060—1061).—Surface tensions of fatty acids in benzene solution in contact with dilute soda solutions have been determined by the drop method. An equimolecular mixture of stearic and myristic acids has the same refractive index as palmitic acid, but they may be differentiated readily by surface tension measurements. An equimolecular mixture of stearic and palmitic acids has a different surface tension from daturic acid (cf. Gerard, B., 1890, 1137). L. F. Hewitt.

Patent.

Refining or separating volatile constituents from materials. [Deodorising oils.] H. M. Lamy-Torrilhon (E.P. 214,273, 12.4.24. Conv., 14.4.23).—The oil or other material is treated with steam or other vapour in separate refining chambers which may be arranged in a column. Heat is recovered from the steam or vapour in one of the chambers and used for heating liquid in another chamber on the multiple-effect principle. J. S. G. Thomas.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Comparison of stick-lacs of different origins. A. Tschirch and H. H. Schäfer (Chem. Umschau, 1925, **32**, 309—314; cf. B., 1924, 103).—Proximate analyses of a number of stick-lacs of various origins were made. The moisture content varied from 1·0 to 6·1%, average 2·9%. Lac from *Zizyphus xylopyrus* showed the highest moisture content, and lac from prepared "gala-gala," the lowest. In general, the water content varied directly as the proportion of sugar and nitrogenous matter present and inversely as the content of wax and resin. The lac produced by male insects appeared to contain the greatest proportion of water-soluble constituents and residue insoluble in solvents, but the least wax and resin, the reverse being the case for the lac produced by female insects. A. de Waele.

Effect of yellow and brown iron oxide pigments upon the rate of oxidation of linseed oil. F. H. Rhodes and J. D. Cooper, jun. (Ind. Eng. Chem., 1925, **17**, 1255—1257; cf. B., 1924, 916).—Ochres retard the initial rate of oxidation of linseed oil, the finely-divided French ochre more so than American ochre. Adsorption of the lead drier from the oil by the pigment is also greater with the French ochre. The final rate of oxidation is increased (except with white ochre which contains very little iron oxide), probably due to the interaction of the iron oxide with the acidic substances produced during drying, whereby catalysts are formed which promote oxidation of the oil; the increase is not so great as with some of the red iron oxide pigments. Raw sienna behaves much like American ochre, but burnt sienna does not retard the initial stages of drying much; both pigments adsorb the drier from the oil almost completely. Apparently small amounts of manganese drier are formed from manganese present in the pigment and counteract the effect of removal of the lead drier. Retardation does not occur with umbers and here again manganese drier is formed. "Metallic brown" behaves much like American ochre. D. G. Hewer.

Degree of dispersity of litharge as a criterion of its suitability for use as a drier for boiled oils. F. Hebler (Farben-Ztg., 1925, **31**, 637—639).—The colour of litharge is dependent on its method of manufacture, the degree of dispersity determining its tint in most cases, although the presence of small proportions of red lead may have some effect. A rough criterion of the degree of subdivision may be obtained from the volume-weight, *i.e.*, the number of c.c. occupied by 100 g. Comparison of two samples of litharge of extreme degrees of subdivision (volume-weights 87 and 25 respectively) showed that the more finely-divided litharge yielded a boiled oil giving a greater increase in weight on drying, but no indications appeared of its greater efficacy as a drier or greater ease of solution in linseed oil. A. de Waele.

Patents.

Titanium pigments. C. A. Klein and R. S. Brown (E.P. 243,081, 25.8.24).—A titanium pigment is obtained by fusing together a titanium ore, such as ilmenite or rutile, with a suitable barium compound, *e.g.*, barium carbonate, with or without a flux, such as fluorspar, and a reducing agent, to form a slag of barium and titanium oxides and metallic iron. Alternatively the titanium ore may be heated with barium sulphate and the mixture of barium oxide, titanium oxide, and iron oxide thus obtained, fused with carbon to obtain metallic iron and a slag containing barium oxide and titanium oxide. The metallic iron is removed from the crushed mass, *e.g.*, by magnetic separation, the slag mixed with sulphuric acid to a paste, and heated to form barium sulphate and titanium sulphate. This is formed into a paste with water and added to boiling water in the presence of organic substances, *e.g.*, aldehydes, sugar, starch, or the like, whereby a titanium compound is precipitated on to the barium sulphate and the precipitation of iron is prevented. The precipitate is washed, dried, and heated. A. de Waele.

Acid-resistant shellac substitute. J. R. Kuhn, Assr. to C. Ellis (U.S.P. 1,564,002, 1.12.25. Appl., 12.1.22).—A spirit varnish is obtained by treating a mixture of *m*- and *p*-cresol with $1\frac{1}{2}$ times its weight of sulphur chloride in the presence of a hydrocarbon diluent, whereby a resin is formed in solution, then blowing the solution with heated air at a temperature below 156°, to remove the diluent and hydrochloric acid, dissolving the deodorised resinous product in an alcoholic solvent, and adding a small amount of phosphoric acid sufficient to change the colour of the solution from yellow to brown. A. de Waele.

Preparation of condensation products [artificial resins]. Badische Anilin- u. Soda-

FABR., Assees. of H. RAMSTETTER (G.P. 416,252, 31.1.23).—In the preparation of artificial resins from carbamide or its derivatives and formaldehyde, the condensation is carried out under the influence of radiation of short wave-length, *e.g.*, from a quartz mercury lamp, in the presence or absence of condensing agents. The rays are applied during the whole reaction or a part of it or during the concentration of the reaction solution or the hardening of the product. The latter has increased solidity and hardness. For example a solution of carbamide is treated at 75° with a mixture of 80% formic acid and 30% formaldehyde at 40°, and heated for $\frac{1}{4}$ hr. under reflux, while exposed to the light of a quartz mercury lamp. Excess of formaldehyde is distilled off under reduced pressure at 40° till a syrup remains, and the latter is poured into flat moulds. On keeping it finally forms transparent, flexible plates, the hardening process also being accelerated by exposure to ultraviolet light.

B. FULLMAN.

Preparation of oily and resinous condensation products. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (G.P. 416,904, 17.1.20).—Polynuclear aromatic hydrocarbons (except hydrogenated naphthalenes) and their halogen derivatives are heated with aralkyl halides in the absence of condensing agents (to prevent contamination) and without increase of pressure, using earthenware, porcelain, or enamelled iron vessels. For example, equimolecular proportions of naphthalene and benzyl chloride, heated to 160° under reflux, yield on purification of the product by steam distillation and subsequent distillation at 12 mm., an oil, b.p. 180—210°, solidifying to a grease; an oil, b.p. 210—240°; and a viscous liquid resin. Benzyl chloride heated with α-methylnaphthalene gives a viscous oil; with anthracene and tetrachloronaphthalene, resins.

B. FULLMAN.

Production of viscous or resinous masses. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), E. FREUND, and H. JORDAN (G.P. 417,667, 23.9.21).—On treating halogen derivatives of the type $RCHR^1 \cdot Hal$ with surface catalysts (*e.g.*, hydrated aluminium, magnesium, or magnesium-aluminium silicates, kaolin, fuller's earth, silica, and similar substances) at temperatures below 180°, with or without diluting media, and if necessary under pressure, hydrogen halides are split off and polymerisation takes place, the products being oils of various degrees of viscosity or resins of varying degrees of hardness, according to the temperature and dilution. With aluminium-magnesium silicate, 1:2-dibromo-*ac*-tetrahydronaphthalene yields (at 100°) a resin, and benzyl chloride in xylene yields saturated oils of varying viscosity. Chlorinated decahydronaphthalene, the aliphatic halides from solar oil and "yellow oil," and pinene hydrochloride may also be used.

B. FULLMAN.

Preparation of resinous or plastic masses. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), E. FREUND, and H. JORDAN (G.P. 417,668, 23.9.21; cf. preceding abstract).—In a process similar to that described in the preceding abstract the reaction is carried out in the presence of formaldehyde and its derivatives, or (for plastic masses) other aldehydes, ketones, phenols, and their derivatives (these substances being added before or during the reaction). In the case of phenols and benzyl chloride molecular proportions are not used. The resins prepared according to the method of the preceding abstract soften at 100° or above; addition of formaldehyde to the reaction mixture before or during reaction gives rise to fusible resins. For example, tetrachloronaphthalene (in naphthalene solution) and phenol, heated with ferric oxide, yield a resin which softens at low temperatures; benzyl chloride and paraformaldehyde (with "frankonit") yield a hard, infusible resin; equal weights of benzyl chloride and phenol (with "frankonit") yield a plastic mass; technical xylyl chloride and acetophenone (with "tonsil"), and technical xylyl chloride and camphor (with "frankonit F.C.") yield viscous oils.

B. FULLMAN.

Lithopone product. J. L. MITCHELL (U.S.P. 1,565,185, 8.12.25. Appl., 6.10.22).—See E.P. 209,270; B., 1924, 185.

Purifying waste waters (U.S.P. 1,557,188).—See XXIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Two-phase structure [of rubber]. E. A. HAUSER (Rev. gén. Colloid., 1925, 3, 289—293, 321—324).—A general review of work bearing on the subject.

D. F. TWISS.

Significance of the protein film [around rubber globules]. H. P. STEVENS (Indiarubber J., 1925, 70, 1059—1060).—The results of Sebrell, Park, and Martin (B., 1925, 1000) as to the persistence of the rubber particles during coagulation are in accord with the author's earlier demonstration (Comm. 8th Int. Congr. Appl. Chem., 1912, 9, 17) of the influence of the adsorbed protein film on the behaviour of smoked sheet rubber towards benzene. The protein film in crêpe rubber coagulated in the presence of tannic acid appears to be more resistant to mechanical treatment than that in ordinary crêpe rubber.

D. F. TWISS.

Problems of vulcanisation. IV. Shell aggregation and colloidal crystallisation of caoutchouc. H. FEUCHTER (Koll. Chem. Beihefte, 1925, 21, 171—194; cf. B., 1925, 367).—An elaboration and extension of the earlier theory (*loc. cit.*). The structure of rubber involves a spheroidal system on the surface of which dimethyloctadiene or "polyprene" nuclei are disposed sterically. The elasticity arises from the valency forces between the components of the system, and the phenomena of "grain" and crystallisation in rubber also find explanation. The latex particle is an aggregate of polyprene nuclei forming a "co-molecule" the volume of which is $1 \cdot 6 \times 10^{13}$ times that of the hæmoglobin

molecule; the assumption that the skin and interior of the latex globule form two distinct constituents in rubber is unnecessary. D. F. TWISS.

Action of "vulkone." Reversion [of rubber during vulcanisation]. L. STOLL (Gummi-Ztg., 1925, **40**, 676—678).—When vulcanised over a range of periods by heating with steam at a pressure of 3·2 atm., mixtures of rubber 100, zinc oxide 5, sulphur 5, and an accelerator "vulkone" 0·5—6 pts. give indications of "reversion," particularly with the higher proportions of accelerator. The evidence of "reversion" includes increase in ultimate extension and in rate of swelling in benzene, and decrease in hardness, with progressive "vulcanisation." D. F. TWISS.

Hot vulcanisation in air. B. V. BYZOV (Russian J. Chem. Ind., 1925, **1**, [2], 14—17).—Vulcanisation of rubber in hot air requires the presence, in sufficient proportions, of sulphur and of compounds of lead, mercury, or silver with organic hydroxy-compounds of acid character. After the vulcanisation, the lead compounds are insoluble in chloroform and exist only to a small extent as lead sulphate and sulphide, which are evidently secondary products. T. H. POPE.

Rubber and the rubber industry. J. C. BONGRAND (Chim. et Ind., 1925, **14**, 823—838).

PATENTS.

Treating rubber composition. J. B. DICKSON, Assr. to B. F. GOODRICH Co. (U.S.P. 1,564,050, 1.12.25. Appl., 31.1.23).—A small quantity of an accelerator of vulcanisation is applied to the surface of a rubber mass containing a vulcanising agent, so as to induce a relatively high degree of vulcanisation at the surface. D. F. TWISS.

Vulcanisation of caoutchouc. CHEM. FABRIK KALK G.m.b.H., Assees. of H. OEHME (U.S.P. 1,565,812, 15.12.25. Appl., 13.7.25).—See E.P. 239,173; B., 1925, 932.

XV.—LEATHER; GLUE.

PATENTS.

Producing gas-tanned leather. R. H. PICKARD, D. JORDAN-LLOYD, and A. E. CAUNCE (E.P. 243,089, 27.8.24).—Pelt, hide, or skin is dehydrated with acetone or other volatile solvent of water, the solvent evaporated off, and the dry pelt subjected to the action of a tanning agent in gaseous form, *e.g.*, formaldehyde. D. WOODROFFE.

Production of leather. R. H. PICKARD, D. JORDAN-LLOYD, and A. E. CAUNCE (E.P. 243,090, 27.8.24).—Pelt, hide, or skin is dehydrated by immersion in acetone until it is in equilibrium with a mixture of acetone and water of *d* greater than 0·810, then drained, the acetone evaporated off, and the pelt, hide, or skin subjected to the action of an aqueous solution of a tanning agent (vegetable, chrome, or gaseous). The pelt is instantly penetrated and a saving of time of 75% is effected. D. WOODROFFE.

Preparation of chrome-tanned leather for storage in the crust condition previous to dyeing. R. H. PICKARD, D. JORDAN-LLOYD, and A. E. CAUNCE (E.P. 243,091, 27.8.24).—Wet chrome-tanned leather prepared for dyeing is steeped in, or sprayed with, acetone, then drained, the acetone evaporated off by subjecting the leather to the action of air or inert gas warmed to 57°, and the leather stored. The density of the acetone bath should not rise above 0·810 during use. D. WOODROFFE.

Tanning of leather. L. A. JORDAN (E.P. 243,144, 31.10.24).—A small amount of a synthetic organic dye, *e.g.*, Quinoline Yellow, Quinoline Yellow KT, Disulphine Green, or Neptune Green, resistant to the bleaching action of sulphurous acid or bisulphites, is added to tanning solutions or extracts which ordinarily produce a reddish coloured leather, *e.g.*, quebracho, mimosa, or kahua, in quantity sufficient to compensate or neutralise the red colour. A soluble bisulphite may also be added if desired. D. WOODROFFE.

Organic thiocarbonates (F.P. 563,214).—See XX.

XVI.—AGRICULTURE.

Relation of biological processes to concentration of cations in soils. J. S. BURD (Soil Sci., 1925, **20**, 269—283).—Studies are reported on the effect of time and various treatments on the concentration and composition of the liquid phase of soil from which the original soil solution has been removed by displacement with water (cf. Burd and Martin, B., 1923, 845 A; 1924, 988). When the soils, after initial displacement, were kept under conditions such that biological activities were reduced to a minimum, the total concentration of the liquid phase increased but remained very low. On the other hand, when the soils were kept under aerobic conditions, the concentration of the liquid from later displacements was greatly increased. Biological oxidations resulting in increases in the concentration of nitrates and sulphates tend in general to increase the total concentration of the soil solution and, in effect, determine the concentration of cations as well as of anions. Under conditions favouring reduction processes, there is a decrease of nitrates without an equivalent decrease of cations, the loss being compensated for by increase of hydrogen carbonate ion. C. T. GIMINGHAM.

Carbon and nitrogen transformations in the decomposition of cellulose by filamentous fungi. H. HEUKELEKIAN and S. A. WAKSMAN (J. Biol. Chem., 1925, **66**, 323—342).—Experiments with a species of *Trichoderma* and one of *Penicillium* isolated from soil indicate that these fungi, whether grown in a liquid medium, on sand or on soil, are capable of decomposing cellulose completely to carbon dioxide and water; as much as 45% of the carbon dioxide produced may be utilised by the organism; there is a definite relationship, which varies with the type and

age of the organism, between the carbon and nitrogen assimilated, but in both cases nitrogen is utilised much more readily in the form of ammonia than as nitrate. C. R. HARINGTON.

Hydrogen-ion concentration of soils as affected by carbon dioxide and by the soil-water ratio, and the nature of soil acidity. W. H. PIERRE (Soil Sci., 1925, **20**, 285—305).—Neither the presence of the roots of actively growing plants nor the passage of carbon dioxide through the soil affects the p_H value of the displaced soil solution. The p_H of some soils, especially very acid ones, varies with the soil-water ratio used in making the suspensions; others show no variation. When soils which exhibit this difference are thoroughly leached, they no longer show any such variation. Data are recorded indicating that the hydrogen-ion concentration of a filtered soil extract or displaced soil solution, determined by the colorimetric method, agrees well (provided certain conditions are adhered to) with the figure obtained electrometrically with a suspension of the same soil. C. T. GIMINGHAM.

Influence of the amount and nature of the replaceable base upon the heat of wetting of soils and soil colloids. W. W. PATE (Soil Sci., 1925, **20**, 329—335).—Saturation of a soil, or of colloidal material extracted from it, with a univalent base lowered the heat of wetting. Saturation with a bivalent base had no very marked effect. Comparing the extracted colloids from different soils, it is shown that the heat of wetting can be positively correlated with the amount of replaceable base in the colloid (cf. Bouyoucos, A., 1925, i, 1228). C. T. GIMINGHAM.

Influence of hydration on the stability of colloidal solutions of soils. L. C. WHEETING (Soil Sci., 1925, **20**, 363—366).—When a known volume of a suspension of a soil colloid in water is brought to a temperature of $-4°$, the volume of water which freezes is greater than the original volume measured. Water of hydration held by the colloid becomes free and is frozen. If a salt of strong hydrating and flocculating powers (*e.g.*, aluminium sulphate) be added, the volume of (free) water which can be frozen is further increased. The presence of a salt of low hydrating power and strong deflocculating action (*e.g.*, sodium carbonate), on the other hand, reduces the amount of freezable water. A correlation exists between flocculation effects and the amount of water of hydration of the colloid, which holds more combined water when in a dispersed condition than when flocculated. Flocculation is brought about by any treatment which reduces the water of hydration below a certain point. The measurements were made by the dilatometer method of Bouyoucos. C. T. GIMINGHAM.

Colloid determination in mechanical analysis [of soils]. R. O. E. DAVIS (J. Amer. Soc. Agron., 1925, **17**, 275—279; Chem. Abstr., 1925, **19**, 3339).—Since the absorption of water vapour by soils is proportional to the amount of colloidal material present, the average absorption over 3·3% sulphuric acid being 0·3 g. per g. of colloid, the percentage of colloid in a soil may be thus determined. The separation of the sand sizes is carried out in the usual way, and the silt obtained by difference. A. A. ELDRIDGE.

Biological measurement of the availability of potassium in soils. D. E. HALEY (Pennsylvania Agr. Exp. Sta. Bull., 1925, [188], 1—8; Chem. Abstr., 1925, **19**, 3341).—Soils treated with potassium over a period of years contained no more water-soluble potassium than untreated soils. A. A. ELDRIDGE.

Distribution of calcium cyanamide [mixed] with soil. E. BLANCK and F. GIESECKE (J. Landw., 1925, **73**, 305—316).—Vegetation experiments in soil with oats indicate that the injurious action of calcium cyanamide on germination and plant growth (due to the presence of dicyanodiamide) is much reduced if the fertiliser is first mixed with a quantity of soil and kept for some days before application. A proportion of the nitrogen is converted into ammonium salts and the percentage utilised by the plants is increased. At the same time, the annoyance caused by the dusty nature of the material when it is being distributed is avoided. C. T. GIMINGHAM.

Influence of irrigation water and manure on the composition of the maize kernel. J. E. GREAVES and D. H. NELSON (J. Agric. Res., 1925, **31**, 183—189; cf. B., 1924, 269; 1925, 415).—Field experiments, extending over 12 years, in which maize was grown on a calcareous soil with and without varying quantities of irrigation water and of farmyard manure, show that the nitrogen content of the grain depends upon the available nitrogen content of the soil. Under irrigation, nitrogen is more rapidly rendered available, but it is also more rapidly removed beyond the reach of the plant. There is an increase of both nitrogen and ash constituents in the grain due to the manure; nitrogen is decreased and ash constituents are increased by the application of irrigation water. The results are in accord with the view that the increased ash and mineral constituents of the grain are due to increased bacterial activity which increases the available plant food in the soil. C. T. GIMINGHAM.

Hop investigations. Manuring experiments, 1924. A. H. BURGESS (J. Inst. Brew., 1925, **31**, 609—612; cf. B., 1924, 956).—The average hop crops obtained during the past three years from the series of plots at Chilham under treatment with various fertilising elements, bear the following relationships: complete manure (including dung) 100·0; complete manure (mineral) 95·2; without phosphate 92·1; without nitrogen 60·1; without potash 54·4; unmanured 40·7. The corresponding values for the Horsmonden plots were: complete manure 100·0; without potash 96·3; without phosphate 92·1; complete manure (including dung) 81·4; unmanured 61·9; without nitrogen 60·9. The differences in resin content were small; the average soft resin percentage corresponding with the various manurial treatments decreased in the same order as the average crop from these plots decreased. C. RANKEN.

Combined lead arsenate and lime-sulphur spray. R. W. THATCHER and L. R. STREETER (New York Agr. Exp. Sta. Bull., 1924, [521], 3—20; Chem. Abstr., 1925, **19**, 3558).—Increase in insoluble material and soluble arsenic in combined lead arsenate and lime-sulphur sprays, yielding a less fungicidal and more harmful mixture, is prevented by the addition of calcium hydroxide (5 lb. per 100 gals.) or casein. A. A. ELDRIDGE.

Influence of soil, season, and manuring on barley. LANCASTER.—See XVIII.

PATENTS.

Manufacture of superphosphates. A. C. HYDE (E.P. 243,192, 19.1.25).—Phosphatic rock, ground fine enough to pass through a 200-mesh sieve, is mixed as a dust cloud, as nearly as possible in the correct proportions, with a fine spray of ordinary concentrated sulphuric acid. Reaction is almost instantaneous and the product can be continuously removed, no drying being necessary. C. T. GIMINGHAM.

Preparation of copper liquors for combating vine-pests. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (G.P. 416,899, 10.2.20).—Addition of sodium or calcium salts of aromatic sulphonic acids, such as benzylanilinesulphonic acid or naphthalenetrisulphonic acids, to the calcium hydroxide liquor or the copper sulphate solution, or to the reaction product or to a copper-sodium carbonate liquor, causes an increase in degree of dispersion, of viscosity, and of adhesive power. With a liquor containing 0·3% of copper salt in the presence of a sulphonate, the results obtained in protection against *Peronospera* are equal to, or better than, those given by similar liquors, without sulphonates, containing 1%, 1·5%, and 2% of copper salt respectively in the first, second, and third sprayings. B. FULLMAN.

Material for combating pests. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING, Assees. of A. STEINDORFF, K. PFAFF, and H. MEYER (G.P. 417,041, 27.5.22).—Naphthenates of bases, such as pyridine, piperidine, or nicotine, which are themselves insecticides, may be used, alone or combined with other materials, against plant lice, caterpillars, and sawflies and their larvæ, and against human parasites. When used in plant sprays the adhesive power of the materials is increased by addition of soaps or soluble resins. B. FULLMAN.

Increasing wetting power of spray mixtures for plants. SOC. CHIM. DES USINES DU RHÔNE (F.P. 562,213, 4.4.22).—A solution of the active agent is treated with a silicic acid sol, or the latter is produced in the solution by the decomposition of alkali silicates with mineral acids or acid salts. For example, a solution of potassium dichromate is treated with sodium silicate solution of 30° B. (*d* 1·26), 10% sulphuric acid is added, with stirring, and the mixture kept for 14 hrs.; or a solution of sodium silicate is treated with sodium hydrogen carbonate, and, after keeping 2—3 days, potassium permanganate is added; or copper sulphate solution is treated with sodium silicate and sodium hydrogen carbonate, and after 1—2 days sodium carbonate is added. The process is specially useful in cases where the active substance is decomposed in presence of organic protective colloids. B. FULLMAN.

Organic thiocarbonates (F.P. 563,214).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Catalysis [precipitation of lime] in the sugar industry. E. SAILLARD (Planter and Sugar Mfr., 1925, **74**, 269; Chem. Abstr., 1925, **19**, 3610).—Precipitation of calcium oxide by sulphurous acid in syrups is incomplete even after 24 hrs.; precipitation by sodium carbonate is more efficient, but not complete. It is accelerated by heat, agitation, or kieselguhr. A. A. ELDRIDGE.

[Determination of sucrose in molasses.] E. SAILLARD (Planter and Sugar Mfr., 1925, **75**, 68—69; Chem. Abstr., 1925, **19**, 3610).—To obtain a true sucrose content value for exhausted molasses, the neutral double polarisation is corrected by subtracting the content of hydrated raffinose from the Clerget readings. A. A. ELDRIDGE.

PATENT.

Manufacture of soluble-starch products. R. HALLER, Assr. to CHEM. FABR. PYRGOS (U.S.P. 1,594,955, 8.12.25. Appl., 24.2.25).—See E.P. 229,623; B., 1925, 896.

XVIII.—FERMENTATION INDUSTRIES.

Influence of soil, season, and manuring on the 1924 barley crop as indicated by the malts made therefrom. H. M. LANCASTER (J. Inst. Brew., 1925, **31**, 601—608; cf. B., 1925, 518).—The barleys were malted for the purpose of determining whether their commercial value was reflected in the resulting malts, both barleys and malts being assessed on an open market basis. In some cases the market value of the barley was too high, in others too low, and the valuations of the barleys were borne out by the malts in six cases only. In an appendix, tables are given by H. L. Hind incorporating extensive analyses of the barleys and malts dealt with in the report. Particulars of the manuring of the experimental plots are also included. C. RANKEN.

[Hops.] Report on work at [Institute of Brewing's] experimental oast, 1924. A. H. BURGESS (J. Inst. Brew., 1925, **31**, 613—622; cf. B., 1924, 958).—For a given temperature, the time of drying cannot be reduced below a certain minimum, however great the air speed and however shallow the layer of hops. The extra time of drying above this minimum varies directly as the depth of loading; an empirical formula is deduced from the results, connecting time of drying with depth of loading and air

speed, under the conditions of the experiments. The time of drying per pound of green hops decreases as the depth of loading is increased; this decrease becomes small when a certain depth is reached, which depends on the air speed used. The rate of loss of water from the hops is rapid at first, but during the last two hours little water is lost. Early application of sulphur produced the best coloured samples. Its effect in restoring the colour of brown hops is very slight, even when large quantities of sulphur are burned. Definite conclusions cannot be reached with regard to the influence of the effect of drying temperature on the brewing value of the hops. In seven different commercial kilns the air velocities varied from about 8 to 20 ft. per minute. A comparison of open and closed fires is made.

C. RANKEN.

Separation of the enzymes of barley malt. H. PRINGSHEIM, A. GENIN, and R. PEREWOSKY (Biochem. Z., 1925, **164**, 117—125).—Separation of the enzymes (diastase, the polyases lichenase and mannanase, the disaccharases cellobiase and mannobiase, maltase) of germinating barley was attempted both by allowing the mixture of enzymes to remain in a solution of such p_H that one group was inactivated more rapidly than another group, and by repeated adsorption on kaolin or aluminium hydroxide from solutions of different p_H with subsequent elution by a buffer solution. After keeping for 18 days at p_H 3—4 mannanase and cellobiase are inactivated but amylase retains slight activity. At p_H 6—7 all the enzymes are more resistant and separation is impossible. Adsorption on kaolin at p_H 3 is quantitative, at p_H 5 less vigorous, and at p_H 8 a difference is obtained in the adsorption of the polyases and disaccharases which is intensified in alcoholic solution. From alcohol of 20% and higher concentrations mannobiase and a small quantity of cellobiase are adsorbed whilst mannanase and lichenase remain in alcoholic solution. By treating mannan with mannanase thus freed from mannobiase, mannobiose was obtained and isolated as the phenylhydrazone. Malt amylase is adsorbed from acid but not from neutral or alkaline solution. Amylase obtained by repeated adsorption from 40% alcoholic solution is completely free from maltase and reducing substances.

P. W. CLUTTERBUCK.

Takadiastase. S. NISHIMURA (Chem. Zelle u. Gewebe, 1925, **12**, 202; Chem. Zentr., 1925, II., 2212).—Takadiastase contains the enzymes, amylase, invertase, maltase, proteases, catalase, lipase, rennin, lactase, inulase, sulphatase, and amidase, of which amylase, invertase, maltase, proteases, catalase, and lipase are quantitatively adsorbed by colloidal alumina at p_H 6·0. If the enzymes are extracted from the alumina by means of phosphate mixture at p_H 8·0, the enzymic activity of the solution so obtained is three times that of the original solution. The individual enzymes differ neither in their capacity to be adsorbed nor in the amounts in which they are extracted. If the solution of extracted enzymes is again brought to p_H 6·0 and the process of adsorption and extraction repeated, the activities of the enzymes then obtained differ markedly. Compared with those of the original solutions, the values corresponding to their activities are: amylase 7·0; invertase 4·63; maltase 5·05; proteases 5·53; catalase 10·0.

C. RANKEN.

Abnormal wines. FONZES-DIACON (Ann. Falsif., 1925, **18**, 532—536).—A weak wine may be considered abnormal but natural when the following four conditions obtain. The sum of the fixed acid and alcohol is less than 12 g. per litre; the tartaric acid index (*i.e.*, the ratio of cream of tartar corresponding to the total tartaric acid to that corresponding to the total potassium) is less than 1; the total potassium in the tartrate is above 4 g. per litre, and the volatile acidity less than 0·7 g. per litre. Wines that have become abnormal owing to the developement of "tourne" diminish in fixed acidity and increase in volatile acidity (above 1), but fulfil the first three conditions given above.

D. G. HEWER.

Oxidisability of iron in wines. J. WOLFF and L. GRANDCHAMP (Compt. rend., 1925, **181**, 939—941).—Wines normally contain iron in the ferrous condition but in certain diseased conditions discoloration occurs owing to the formation of ferric tannate. Certain oxydase-containing fungi, *e.g. Russula delica*, rapidly oxidise the ferrous iron in wines to the ferric condition, the transformation being inhibited by the presence of a minute amount of sulphur dioxide.

L. F. HEWITT.

Composition of the yellow oil obtained in the manufacture of *n*-butyl alcohol by fermentation. C. S. MARVEL and A. E. BRODERICK (J. Amer. Chem. Soc., 1925, 47, 3045—3051).—*n*-Butyl alcohol, active amyl alcohol, *iso*amyl alcohol, *n*-hexyl alcohol and the *n*-butyric, caprylic, and caproic esters of these alcohols were identified in the high-boiling material obtained as a by-product in the *n*-butyl alcohol fermentation. *a-Mono*-n-*amyl* 3-*nitrophthalate*, m.p. 132—133°, and *a-mono*-n-*hexyl* 3-*nitrophthalate*, m.p. 121—122°, are described.

F. G. WILLSON.

Decomposition of cellulose by fungi. HEKUELEKIAN and WAKSMAN.—See XVI.

Hop manuring experiments. BURGESS.—See XVI.

By-product-yeast as source of vitamin-B. HEPBURN.—See XIX.

Mobile-scale densimeters. SETTIMJ.—See XIX.

Dehydration of aqueous alcohol. BARBAUDY.—See XX.

PATENT.

Utilising grape residues (F.P. 590,739).—See II.

XIX.—FOODS.

Use of sodium nitrite in curing meats. W. L. LEWIS, R. S. VOSE, and C. D. LOWRY, JUN. (Ind. Eng. Chem., 1925, **17**, 1243—1245).—Pork hams, beef tongues, and beef hams were cured quite satisfactorily in a pickle in which sodium nitrate

was replaced by one-tenth of its weight of sodium nitrite. Analyses of nitrate and nitrite pickles during use showed that the losses in salt and sugar are approximately the same in both cases, and the final proportions of sodium nitrite in the meat cured with nitrite and nitrate respectively were as follows :—Pork hams, 40, 50 ; beef tongues, 45, 70, and beef hams, 13, 13 p.p.m. Colour fixation proceeded more rapidly with the nitrite cure. Sodium nitrite was also successfully used for dry curing. D. G. Hewer.

Reduction test of milk and its theoretical basis. C. Barthel (Svensk Kem. Tidskr., 1925, **37**, 157—165 ; Chem. Zentr., 1925, II., 2111).—The reduction of methylene-blue by milk is correlated with the bacterial content of the sample. The bacteria consume dissolved oxygen and reduce the colouring matter. Some decolorisation does, however, occur in milk containing only few bacteria ; this is shown to be due to the reducing action of citric acid, which may be present in milk up to 0·25%. In spite of this complication, the test can be made use of by noting the velocity with which reduction takes place. 20 c.c. of milk are mixed with an alcoholic solution of methylene-blue, warmed to about 35°, and the time taken for decolorisation is observed. C. T. Gimingham.

Mobile-scale densimeters. (Modification of Quevenne's lactometer.) M. Settimj (Annali Chim. Appl., 1925, **15**, 378—386).—In the case of both full-cream and separated milks, the density of a sample at any given temperature between 0° and 30° is a function (independent of the temperature) of the density of the sample at 0°. The same holds less accurately for alcohol–water mixtures between the limits of 50% and 75% of alcohol. It is therefore possible to construct a mobile-scale hydrometer which will give the densities of such mixtures directly at any temperature between 0° and 30°. Its stem consists of two parts, the lower one of which slides within the upper and is calibrated. The instrument gives densities directly for the temperature corresponding to the calibration at which the end of the upper part of the stem is set. W. E. Ellis.

Measurement of the hydrogen-ion concentration of cheese by means of the quinhydrone electrode. S. Knudsen (Z. Unters. Nahr. Genussm., 1925, **50**, 300—306).—A method by which the p_H value of cheese may be determined with accuracy by means of the quinhydrone electrode is described. C. T. Gimingham.

Organic acids of tomatoes, particularly citric acid and its state of combination. A. Bornträger (Z. Unters. Nahr. Genussm., 1925, **50**, 273—300).—Tomato fruits in sound condition always contain citric and malic acids in appreciable amounts. Oxalic, tartaric, racemic, benzoic, and lactic acids cannot be detected, nor are volatile organic acids present. Benzoic acid was found in over-ripe fruit. Special attention is given to the question of oxalic acid ; it is never present in the fruits at any stage. Citric acid occurs as primary or secondary citrates, never as a normal salt ; a little free acid may also be present, amounting to not more than 0·05%. The content of citric acid in the juice, in general, falls with complete ripening, and both citric and malic acids disappear when the fruit becomes rotten. The phosphate content also decreases with ripening. Experimental methods are given in detail. C. T. Gimingham.

Manganese chlorosis of pineapples. M. O. Johnson (Hawaii Agr. Exp. Sta. Bull., 1924, [52], 1—38 ; Chem. Abstr., 1925, **19**, 3559).—In experiments with rice and nutrient solutions, manganous sulphate or manganese dioxide caused chlorosis when a normal amount of iron was also supplied, but a large increase in the latter nullified the effect, which is therefore due to a depression of the assimilation of iron, or a deficiency of iron in the plant. Stimulation of plant growth by manganese was not observed, unless an undesirable excess of iron was originally present. The manner in which manganese induces chlorosis is explained by the varying solubility of iron at different hydrogen-ion concentrations. The application of iron salts to the leaves of pineapple plants on manganiferous, acid Hawaiian soils results in the immediate resumption of normal growth. A. A. Eldridge.

Effect of fermentation with specific micro-organisms on vitamin-C content of orange and tomato juice. S. Lepkovsky, E. B. Hart, E. G. Hastings, and W. C. Frazier (J. Biol. Chem., 1925, **66**, 49—56).—Prolonged fermentation of tomato juice with organisms present in fermented maize, under strictly anaerobic conditions, resulted in practically no destruction of vitamin-C ; the absence of the latter from maize silage and from sauerkraut is therefore ascribed to destruction by oxidation, and not by bacterial action. C. R. Harington.

By-product yeast [as source of vitamin-B]. J. S. Hepburn (J. Franklin Inst., 1925, **200**, 767—770).—Feeding experiments on albino rats showed that dried by-product yeast was a source of water-soluble vitamin-B. C. Ranken.

Cocoa. I. Detection of shell. W. Plücker, A. Steinruck, and F. Starck (Z. Unters. Nahr. Genussm., 1925, **50**, 307—315).—Data obtained by determinations of the crude fibre and phosphoric acid in cocoa and chocolate give no reliable indication of adulteration with cacao bean shell. The composition of the shell of beans from different sources varies greatly. Further, adulteration with shell cannot be satisfactorily recognised by microscopical examination unless an actual count of the number of "stone-cells" present is made. A method for this determination is suggested. C. T. Gimingham.

Formation of jellies. A. Mehlitz (Konserven-Ind., 1925, **12**, 467—470 ; Chem. Zentr., 1925, II., 2110 ; cf. B., 1925, 687).—The influence of the amounts of dry matter (sugar), pectin, and acid (in apple pulp) on jelly formation has been investigated. The formation of a jelly occurs only when

the content of dry matter lies between 50 and 70%. With 50% of dry matter, at least 1·2% of pectin is required, this amount being reduced to half with 70% of dry matter. The optimum p_H for jelly formation is 2·9—3·1. C. T. GIMINGHAM.

Degree of sweetness of saccharin and dulcin. TÄUFEL and KLEMM.—See XX.

PATENTS.

Bleaching nuts. A. W. CHRISTIE, Assr. to CALIFORNIA WALNUT GROWERS' ASSOC. (U.S.P. 1,558,963, 27.10.25. Appl., 30.4.25).—The nuts are immersed first in a solution in which nascent chlorine is being liberated, and then in sulphurous acid. D. G. HEWER.

Concentrating and drying fruit juices. W. H. DICKERSON, Assr. to INDUSTRIAL WASTE PRODUCTS CORP. (U.S.P. 1,562,309, 17.11.25. Appl., 2.8.22).—Fruit juices are concentrated by freezing to preserve aromatic ethers, and then dried to a powder by spraying into a hot gas. C. RANKEN.

Treatment of chocolate. J. B. BARNITT (U.S.P. 1,563,682, 1.12.25. Appl., 26.7.22).—Ground roasted cacao nib from which a large proportion of the cacao butter has been removed is bleached in a slightly alkaline solution of hydrogen peroxide. D. G. HEWER.

Treating milk. A. W. BOSWORTH and L. H. CHRYSLER (U.S.P. 1,563,891, 1.12.25. Appl., 20.12.24).—A substance containing the phosphoric acid radical is added to the milk in such quantity that the milk is rendered slightly alkaline and the calcium is converted into insoluble calcium phosphate, whilst the casein is left in solution. The precipitate is removed, and any undesired excess of alkali in the milk is then neutralised. D. G. HEWER.

Manufacture of bran food. J. L. KELLOGG, Assr. to KELLOGG CO. (U.S.P. 1,564,181, 1.12.25. Appl., 26.10.22).—A mixture of bran and an agent containing diastatic enzymes is moistened and heated to convert the bran starches into maltose, and the product is cooked, dried, disintegrated, and toasted. D. G. HEWER.

Recovering oil or fat from milk and cream. A. E. WHITE. From MILK OIL CORP. (E.P. 243,792, 30.11.23).—See U.S.P. 1,485,700 and 1,485,702; B., 1924, 397.

Treating [bleaching] flour, meal, or milling products. T. KROEBER, Assr. to N. V. NOURY & VAN DER LANDE HANDELMAATSCHAPPIJ (U.S.P. 1,565,375, 15.12.25. Appl., 29.4.25).—See E.P. 213,268; B., 1924, 824.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Roots of *Gentiana lutea*, L. R. BINAGHI and P. FALQUI (Annali Chim. Appl., 1925, 15, 386—396).—Deprived of its branches and aerial parts, and freed from soil, the root was dried in air at the ordinary temperature. Its percentage composition was as follows:—Moisture, 13·78; ash, 6·21; alcohol-soluble matter, 52·00; residue, 28·01. From the fresh, pulverised root the following compounds were extracted by a modification of Tanret's method (J., 1905, 1123, 1124): a waxy substance, m.p. 142—143°, $[\alpha]_D^{20}$ —29·4° (in chloroform), resembling Tanret's gentiostearin; resins; gentiopicrin, which hydrolysed to dextrose and gentiogenin; gentiamarin and gentiin (Tanret, *loc. cit.*); gentisein, m.p. 266—267°; sucrose and gentianose (Nordan and Bourquelot, A., 1898, i., 349). Having regard to their relatively high content of extractive matter, and of the active principle, gentiopicrin, the roots should be of value for pharmaceutical purposes. W. E. ELLIS.

Industrial preparation of sodium methylarsinate and derived salts. (Miss) M. G. TAVERNARI (Boll. chim. farm., 1925, 64, 609—612).—A convenient method and apparatus are described for the preparation, in 1·75-kg. lots, of commercially pure methyl iodide and for the conversion of this into sodium (or potassium) methylarsinate by treatment with arsenious anhydride and sodium (or potassium) hydroxide under pressure. The iron salt may be obtained by evaporating on a glass plate a mixture of green iron citrate and sodium methylarsinate in equal weights. T. H. POPE.

Natural and artificial sweetening materials. I. Degree of sweetness of saccharin and dulcin. K. TÄUFEL and B. KLEMM (Z. Unters. Nahr. Genussm., 1925, 50, 264—273).—The degree of sweetness (cf. Paul, B., 1921, 601 A) of both saccharin and dulcin (*p*-phenetole-carbamide) in water or water-alcohol solution is dependent on the concentration. Curves and formulæ are discussed from which the composition of the optimum mixtures of these two substances can be calculated. C. T. GIMINGHAM.

Manufacture of calcium citrate and citric acid from lime juice. F. H. S. WARNEFORD and F. HARDY (Ind. Eng. Chem., 1925, 17, 1283—1286).—High-grade calcium citrate yielding pale-coloured citric acid solutions from which white crystals are deposited can be obtained by treating the defecated juice at ordinary temperature with a quantity of a thin cream prepared from fine-grained lime sufficient to neutralise the total acid present, and then boiling. The calcium citrate is collected, washed with hot water, and worked up as usual. Alternatively the defecated juice may be partly hot-limed (to p_H 4·0), treated with "norit," and liming completed; or, the defecated juice may be treated with sufficient sodium carbonate at ordinary temperature to neutralise one-third of the total acidity (p_H about 3·8), 1% of "norit" added, the mixture boiled for 15 mins., cooled, and filtered, and the cake of "norit" washed with cold water. Any turbidity may be removed by "filter-cel." Calcium chloride is then added to the liquid in excess of the amount equivalent to the sodium carbonate, the mixture boiled, and

neutralisation completed with milk of lime, followed by a small excess of calcium carbonate. After again boiling, the precipitated calcium citrate is filtered off and washed, and finally converted into perfectly colourless citric acid. A high yield is obtained with the use of less lime and sulphuric acid than usual, although with the additional cost of soda ash and calcium chloride. D. G. HEWER.

Dehydration of aqueous alcohol by rectification with benzene. J. BARBAUDY (Compt. rend., 1925, **181**, 911—913; cf. B., 1925, 608).—The phase-rule diagram for the alcohol-benzene-water system is described, and is utilised for the elucidation of Young's method of dehydrating alcohol (cf. B., 1902, 721). S. K. TWEEDY.

Determination of arsenic and silver in silver arsenobenzenes. U. CAZZANI (Boll. Chim. farm., 1925, **64**, 513—515; cf. B., 1919, 792 A).—In order to determine the arsenic, 0·20 g. of the substance mixed with 1 g. of finely powdered potassium permanganate is treated with 5 c.c. of 30% sulphuric acid and then, shaking continually, with 10 c.c. of the concentrated acid. After keeping some minutes, the mixture is treated with hydrogen peroxide till colourless and diluted with 30 c.c. of water. It is then heated cautiously until sulphuric acid commences to be evolved; it is cooled, diluted with 40 c.c. of water, 2 c.c. of hydrochloric acid are added, and the solution is filtered. The precipitate is washed with 10—15 c.c. of water. To the cold filtrate are added 2·50 g. of potassium iodide, and the iodine set free after 1 hr. is titrated with standard thiosulphate solution. The silver is determined by mixing 0·30 g. of the substance with 2 g. of finely powdered potassium permanganate, treating with 10 c.c. of 30% sulphuric acid, and finally with 10 c.c. of the concentrated acid. After occasional shaking during 15 min., the mixture is diluted with 50 c.c. of water; ferrous sulphate is added until the liquid is yellowish and it is then titrated with ammonium thiocyanate. By the following method both metals are determined simultaneously: the substance (0·40 g.) mixed with 3 g. of powdered potassium permanganate is treated with 10 c.c. of 30% sulphuric acid followed by 20 c.c. of the concentrated acid. After occasional shaking during 15 min., the mixture is treated with sufficient hydrogen peroxide to remove the excess of potassium permanganate and manganese dioxide. The liquid is diluted with 40 c.c. of water and heated until sulphuric acid begins to be evolved. It is then cooled, diluted with 60 c.c. of water, and made up to 100 c.c. To 50 c.c. of the solution, 2 c.c. of hydrochloric acid are added; the precipitate is collected and washed. The filtrate is treated with 2 g. of potassium iodide and titrated after 1 hr. with 0·1*N*-thiosulphate. To the other 50 c.c., 5 c.c. of saturated ferric alum solution are added; after acidifying with nitric acid, it is titrated with 0·1*N*-thiocyanate. W. E. ELLIS.

Nepetella, pennyroyal, and origanum oils. G. ROMEO and U. GIUFFRÈ (Annali Chim. Appl., 1925, **15**, 363—373; cf. Pellini, B., 1923, 858 A).—The analytical results of earlier workers, of which full references are included, are compared with the authors', from which the following data are taken:—Nepetella oil (*Calamintha nepeta*, var. *canescens*, *Satureja nepeta*): d^{15} 0·9227, α_D^{20} + 14·33°, n_D^{25} 1·4792, pulegone 49%. Pennyroyal oil (*Mentha pulegium*, var. *hirsuta*): d^{15} 0·9392, α_D^{20} + 26·5°, n_D^{25} 1·4802, pulegone and piperitone 66%. Origanum oil (*Origanum virens*): d^{15} 0·9226, α_D^{20} — 2·10°, n_D^{25} 1·4943, phenols (thymol and carvacrol), 45%. W. E. ELLIS.

Saffron. G. PIERLOT (Chim. et Ind., 1925, **14**, 839—850).

See also A., 1926, 18, **Influence of traces of water on solubility of salicylic acid in benzene** (COHEN and VAN DOBBENBURGH). 21, **Colloid chemistry of bismuth and its compounds** (KUHN and PIRSCH). 48, **Hydrogenation of aldehydes and ketones** (FAILLEBIN). 51, **Sugars [preparation of saccharic acid, *l*-arabonic acid, etc.]** (KILIANI). 56, **Synthesis of *p*-cymene from *iso*propyl alcohol. Syntheses with magnesium *p*-*iso*propylphenyl bromide** (BERT). 60, **Relation between chemical constitution and pungency in acid amides** (JONES and PYMAN). 63, **Esters and sulphonic acids of anthranilic and methylanthranilic acids** (KELLER and SCHULZE). 65, **Decarboxylation of tyrosine and leucine** (WASER). 72, **Constitution of pulegone** (GRIGNARD and SAVARD); **Oil of flowering *Tagetes glandulifera*** (JONES and SMITH); **Action of oxalic acid on terpin hydrate. Reaction of Japanese acid clay to terpin hydrate and terpineol** (ONO); **Occurrence of sylvestrene** (RAO and SIMONSEN).

Oils of chaulmoogra group ANDRÉ.—See XII.

PATENTS.

Manufacture of alkamine esters of *N*-substituted *p*-aminobenzoic acids. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 241,767, 15.1.25; cf. E.P. 17,162 of 1905, B., 1906, 607).—New surface anæsthetics, superior to cocaine in activity, are prepared by exchanging the hydrogen atom of the carboxyl group in *p*-aminobenzoic acid for an alkamine residue, and a hydrogen atom of the amino-group for an alkyl- or alkyloxyalkyl-residue containing more than two carbon atoms. They may be obtained by esterifying an *N*-monoalkyl or *N*-monoalkyloxyalkyl derivative of *p*-aminobenzoic acid with an amino-alcohol or by alkylating an alkamine ester of *p*-aminobenzoic acid. For example, equimolecular quantities of *p*-*N*-propylaminobenzoic acid, m.p. 165—168° (prepared by Houben and Freund's method, cf. A., 1914, i, 43), and β-diethylaminoethyl alcohol are mixed with 10 times their joint weight of toluene. The mixture is saturated with hydrogen chloride and heated for about 8 hrs. at 150°, while a current of hydrogen chloride is slowly passed. The product, *β-diethylaminoethyl* p-N-*propylaminobenzoate*, is an oil, b.p. 217°/7 mm.; the *monohydrochloride* has m.p. 103—104°. The same compound may also be obtained

by condensing β-diethylaminoethyl *p*-aminobenzoate with propyl bromide in propyl alcohol. The preparation of the following compounds is described :— *β-diethylaminoethyl* p-N-*β-methyoxyethylaminobenzoate*, an oil, b.p. 245°/9 mm. (*hydrochloride*, crystals, m.p. 86°) from *p*-*N*-β-methoxyethylaminobenzoic acid, m.p. 159—161° and ethylene chlorohydrin, or from β-diethylaminoethyl *p*-aminobenzoate, β-chloromethyl ethyl ether and ethylene glycol; *β-diethylaminoethyl* p-N-*allylaminobenzoate*, a yellowish oil, b.p. 232°/10 mm. from ethyl *p*-*N*-allylaminobenzoate, m.p. 60—62° (free acid has m.p. 144—146°), and β-diethylaminoethyl alcohol; *γ-diethylaminopropyl* p-N-*propylaminobenzoate* (*monohydrochloride*, m.p. 113°) from ethyl *p*-*N*-propylaminobenzoate, m.p. 70°, and γ-diethylaminopropyl alcohol; *β-diethylaminoethyl* p-N-*isoamylaminobenzoate*, a light brown oil, from ethyl *p*-*N*-*iso*amylaminobenzoate, m.p. 68° (cf. Houben and Freund, *loc. cit.*) and β-diethylaminoethyl alcohol; *β-piperidinoethyl* p-N-*propylaminobenzoate*, m.p. 157—158°; and the *monohydrochloride* of *β-piperidinoethyl* p-N-*methoxyethylaminobenzoate*, m.p. 138°, from ethyl *p*-*N*-β-methoxyethylaminobenzoate, b.p. 199—200°/8 mm. and β-piperidinoethyl alcohol. A. COULTHARD.

Preparation of substances to be taken internally by a patient before radiographic examination. T. THORNE-BAKER (E.P. 241,968, 23.6.24).—Salts of heavy metals opaque to *X*-rays are brought into a very fine state of division and emulsified with gelatin by treating a solution of a compound of the heavy metal with an excess of a soluble non-poisonous salt which has the capacity of precipitating the desired metal salt but does not precipitate the gelatin. The gelatinous product, after washing, is physically similar to a photographic emulsion and can be administered with ordinary food in the form of jellies or otherwise. For example, a solution of 20 pts. by weight of barium chloride, 2 pts. of gelatin, and 50 pts. of water at 43° is added slowly to a solution (also at 43°) of 13 pts. of ammonium sulphate, 1 pt. of glycerin, and 30 pts. of water while stirring energetically. The product is stabilised, if necessary, by the addition of more gelatin. After the jelly has set, it is shredded, washed, and dried. A. COULTHARD.

Preparation of ethylene glycol from ethylene oxide. CHEM. FABR. KALK G.M.B.H., Assees. of H. OEHME (G.P. 416,604, 15.8.23).—Ethylene glycol solution is produced from ethylene oxide and very dilute sulphuric acid, and is removed, as formed, from the sphere of reaction. For example, a reaction vessel connected by an overflow tube with a still, is filled almost up to the overflow tube with faintly acid water. Ethylene oxide is then passed in by another tube, and, by increasing the volume of the acid solution, drives dilute ethylene glycol continuously into the still, where it is concentrated out of contact with the oxide, the evaporated water being condensed and returned to the first vessel. Occasional small additions of sulphuric acid must be made. No polyglycols are formed. B. FULLMAN.

Preparing saturated aldehydes from unsaturated aldehydes. BADISCHE ANILIN- U. SODA-FABRIK (G.P. 416,906, 29.3.23. Addn. to 350,048). —Unsaturated aldehydes are treated in the presence of the copper catalyst, described in the chief patent (cf. U.S.P. 1,410,223; B., 1922, 347 A; also G.P. 407,837; B., 1925, 475), with hydrogen insufficient for the complete formation of saturated alcohols; the latter, if formed, are easily separated from the saturated aldehydes. The results are better than those obtained with nickel catalysts. For example, 100 c.c. of crotonaldehyde vapour passed over finely divided copper, with 30 litres of hydrogen, at 170°, yield a product containing 80% of butaldehyde and 20% of crotonaldehyde; using 50 litres of hydrogen at 180°, the product contains 60% of butaldehyde and 40% of *n*-butyl alcohol. B. FULLMAN.

Preparation of ethyl chloride from ethylene. CHEM. FABR. VORM. WEILER-TER MEER (G.P. 417,170, 24.2.24).—The reaction between ethylene and hydrogen chloride is catalysed by metal chlorides or their derivatives (*e.g.*, ferric, aluminium, nickel, or cobalt chloride, or the aluminium chloride–ethylene compound), loss of activity of the catalyst being compensated by treating it with gaseous chlorine, either during the course of the reaction or by treating the exhausted catalyst separately with chlorine at 100°. For example, an equimolar mixture of ethylene and hydrogen chloride is passed, simultaneously with chlorine (5% by vol. of ethylene), over the aluminium chloride–ethylene compound at 80°. The chlorine is led in by a separate tube, and only meets the ethylene in contact with the catalyst. 43% of the theoretical yield of ethyl chloride is obtained, with no significant formation of dichloroethylene. B. FULLMAN.

Preparation of pure 1-phenyl-2 : 3-dimethyl-5-pyrazolone. K. T. HERBST (G.P. 417,696, 25.8.21).—Solutions of the crude salts of the pyrazolone are treated with sodium perchlorate, if necessary after decolorising with sulphur dioxide. The perchlorate of the base crystallises out (being seeded if necessary), is washed, and decomposed with sodium carbonate. B. FULLMAN.

Calcium preparation [calcium sodium lactate]. H. UMBER, Assr. to J. A. VON WÜLFING (U.S.P. 1,559,478, 27.10.25. Appl., 28.5.23).—Concentrated lactic acid (4 mols.) is mixed with concentrated caustic soda (2 mols.) and quicklime (1 mol.), and the mixture is dried at a low temperature. A double compound of sodium and calcium lactates containing 4 mols. of water is obtained, which is of therapeutic value. T. S. WHEELER.

Prophylactic [mercuriated salvarsan]. O. LOWRY, Assr. to TECHNICAL PRODUCTS CORP. (U.S.P. 1,559,899, 3.11.25. Appl., 20.12.20).—The

dihydrochloride of diaminodihydroxyarsenobenzene is treated in acidified methyl alcohol solution with a solution of mercuric chloride in the same solvent, and the precipitate of "mercuriated" salvarsan is mixed with glycerin, gelatin, and mercuric chloride to give a prophylactic of firm consistency at ordinary temperatures but fluid at body temperatures.

T. S. Wheeler.

Preparation of water-, alcohol-, and oil-soluble tuberculin preparations. Farbw. vorm. Meister, Lucius, u. Brüning, Assees. of L. Lautenschläger and M. Bockmühl (G.P. 417,443, 9.6.23).—The precipitates obtained from liquid culture media on which tubercle bacilli of human or animal origin have been grown, or the bacilli themselves, or parts of them, are treated with alcohols and mineral acids. The products, which are separated by known methods, are probably esters of tuberculinic acid, and are very active. For example, dry tuberculin is suspended in absolute methyl alcohol, a stream of dry hydrogen chloride is led through at 15° until solution occurs, the solution concentrated in a vacuum at 40—50°, the residue freed from hydrogen chloride, and the pure compound precipitated by ether from methyl alcohol solution. Dead tubercle bacilli freed from fat and wax may be esterified with ethyl alcohol, and dry tuberculin with absolute amyl alcohol in the presence of sulphuric acid. The products are very soluble in water, alcohols, glycols, and glycerol, slightly soluble in oils and fats. Owing to their solubility they should penetrate skin more rapidly, and hence be more effective in treatment of skin diseases, than former tuberculin preparations.

B. Fullman.

Preparation of thymol from 2-cymidine [*o*-aminocymene]. G. Austerweil (E.P. 221,226, 30.8.24. Conv., 31.8.23).—See G.P. 413,727; B., 1925, 864.

Producing methyl alcohol and methylene chloride from methane. T. Goldschmidt A.-G., Assees. of J. Weber and P. Erasmus (U.S.P. 1,565,345, 15.12.25. Appl., 8.4.25).—See E.P. 227,475; B., 1925, 784.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photochemistry of photographic films. F. Weigert (Z. Physik, 1925, **34**, 907—917).—The author discusses and tests Eggert and Noddack's hypothesis (cf. A. 1925, ii, 573) that only a small part of the energy incident on a photographic plate is photochemically active, the major part being absorbed by a yellow colouring matter in the gelatin, especially in the case of coarse-grained plates in which the light-path is lengthened by multi-reflections. With normal and fixed plates dyed artificially with a yellow dyestuff (Filter Yellow) not adsorbed by the silver halide grains, the increased absorption due to the dye was even less in the case of the emulsions than with the clear gelatin layers. Any increase in the length of the light-path due to multi-reflections from the grains is therefore unimportant. All conclusions of Eggert and Noddack based on optical absorption by the gelatin are therefore wrong, and these authors have not established the Einstein law for photographic plates.

W. Clark.

Photochemistry of photographic films. J. Eggert and W. Noddack (Z. Physik, 1925, **34**, 918—920).—Weigert's results (cf. preceding abstract) show that a mixture of the three absorbing substances silver bromide, gelatin, and dye in a turbid film absorb less light than the gelatin and dye alone in a clear layer; *i.e.*, turbid films and clear films cannot be considered as similar for purposes of calculation. It is not surprising that Filter Yellow should act differently from the weakly absorbing gelatin under the circumstances. It is possible that in the dried dyed plate the yellow dye is adsorbed by silver bromide, so introducing a further optical complication.

W. Clark.

Photochemical decomposition of silver chloride. Hartung.—See A., 1926, 34.

Application of spectro-photography to measurement of high temperatures. Griffith.—See I.

Patents.

Producing colour pictures. L. E. Taylor, Assr. to Famous Players-Lasky Corp. (U.S.P. 1,560,437, 3.11.25. Appl., 19.6.23).—Two negatives taken through a red and a blue-green screen respectively are developed and immersed in a solution of hydrogen peroxide, cupric sulphate, and sulphuric acid. The gelatin is removed from the film base by this solution to a degree depending on the exposure. The negatives are dried and that taken through the blue-green screen is immersed in a red dye and that through the red screen in a blue-green dye. On superposition a coloured negative of the original is obtained. Positives are obtained by a similar process.

T. S. Wheeler.

Reversal process for photographic development emulsions. E. O. Langer (G.P. 416,120, 22.7.24).—The metallic silver in the unfixed image is converted into a modification of silver chloride not reducible by developers, by using a bleaching bath containing a trace (up to 1:1000 of the bath) of a suitable metal (chromium, iron, copper, etc.) or metal salt. The presence of organic or inorganic acids in the bleaching bath accelerates bleaching and assists in rendering the silver chloride non-reducible by the second development. Suitable bleaching baths are: (1) potassium chloride, 10 g.; metallic copper, 0·1 g.; formic acid, 5 c.c.; (2) hydrochloric acid, 4 c.c.; sodium dichromate, 0·03 g.; water, 100 c.c.; (3) sodium chlorate, 10 g.; ferric chloride, 0·2 g.; water, 100 c.c.

W. Clark.

Printing kinematograph films. R. Roland and L. Burstein (F.P. 591,806, 21.1.25).—A film is coated with a mixture of hydrocarbons, albumin, and chromium salts, dried, and exposed under the

negative. It is then washed consecutively with water, dilute acid, and water, dyed, and dried.
W. CLARK.

Producing water-insoluble multicoloured [photographic] screens. J. H. CHRISTENSEN (U.S.P. 1,564,202, 8.12.25. Appl., 20.5.24).—See E.P. 216,853; B., 1924, 998.

Producing photographic copies by means of phosphorescent substances. J. H. CHRISTENSEN (U.S.P. 1,565,256, 15.12.25. Appl., 30.7.20).—See E.P. 160,739; J., 1921, 791 A.

XXII.—EXPLOSIVES; MATCHES.

Explosion wave and detonation wave. P. LAFFITTE (Ann. Physique, 1925, [x], 4, 587—694).—A full account of investigations on the explosion of mixtures of carbon disulphide and oxygen, and of some solid explosives (cf. B., 1923, 640 A, 814 A; A., 1924, ii, 399, 580; 1925, ii, 135).
A. B. MANNING.

Assay of potassium chlorate. LEHM and FINK.—See VII.

PATENTS.

Removing tetranitromethane from technical trinitrotoluene. R. GÄRTNER (G.P. 416,905, 1.1.24, and 417,378, 22.1.24. F.P. 590,951, 26.12.24).—In the upper part of a drying-tower, liquid technical TNT is atomised by a hot air-blast, the resulting mist being exposed to a second similar blast to retard solidification of the droplets. The tetranitromethane volatilises and the purified TNT (which has its natural light colour, and is free from all unstable foreign substances, or those injurious to the worker) is cooled and solidified by a cold blast in the lower part of the tower. The atomising hot air-blast (and if desired the other hot air-blast) may be replaced by a steam-blast, in which case there is greater ease in removing the tetranitromethane and in the regulation of the blast by control of the pressure, and the substances volatilised may be more easily recovered.
B. FULLMAN.

Coating for nitrocellulose powder grains. T. L. DAVIES (U.S.P. 1,561,219, 10.11.25. Appl., 28.4.22).—A carbamide with only two alkyl groups symmetrically substituted in the molecule is used as one of the constituents of the coating.
S. BINNING.

Nitrating cellulose. G. JUER, Assr. to TUBIZE ARTIFICIAL SILK CO. OF AMERICA (U.S.P. 1,562,093, 17.11.25. Appl., 5.6.23).—Cellulose and nitrating acids are thoroughly mixed and the partially nitrated product and admixed acids are transferred to a rotary cylinder and mixed until nitration is complete.
J. S. G. THOMAS.

XXIII.—SANITATION; WATER PURIFICATION.

Determination of dissolved oxygen in water in presence of nitrite. M. E. STAS (Chem. Weekblad, 1925, 22, 584—585).—The various methods described in the literature are criticised, and that of Alsterberg (B., 1925, 940) is recommended as most accurate and convenient.
S. I. LEVY.

Loss of nitrogen on purification of sewage water by the activated sludge method. L. CAVEL (Compt. rend., 1925, 181, 1101—1103).—On treatment with 20% of activated sludge, sewage water with added ammonia or ammonium salts loses over 40% of the total nitrogen present. It is suggested that this loss is in the form of free nitrogen. Less nitrogen is probably lost under industrial conditions.
L. F. HEWITT.

Physics and chemistry of gas masks. H. ENGELHARD (Z. Elektrochem., 1925, 31, 590—593).—Gases of an average density of 1·4 compared with air, with molecular radii not exceeding 10^{-5} cm., have an average Brownian movement of 10^{-3} cm. or more per second. In the usual filtering media of pore-diameter 10^{-2} to 10^{-4} cm. they easily reach the walls of the filter in a transit period of 0·1—1·0 second; hence adsorption is favoured. With smoke particles of 10^{-5} cm. diameter or over, the Brownian movement is slower, and it is necessary to superpose several layers of absorbent. When the size of particles exceeds 10^{-5} cm. there is an improvement in absorption owing to increased deposition by gravity, and direct mechanical filtration. The retention of liquid particles by materials which are wetted by the liquid is complete, but for solid particles a material having serrated fibres is required. The efficiency of an absorbent which acts by chemical reaction is enhanced by mixing a highly adsorptive substance with it.
W. A. CASPARI.

PATENTS.

Base-exchange water softener. O. R. SWEENEY, Assr. to WARD-LOVE PUMP CORP. (U.S.P. 1,557,117, 13.10.25. Appl., 9.2.25).—An apparatus for water-softening by the base-exchange process consists essentially of a container for the water-softening material, provided with a diaphragm having extremely fine interstices, *e.g.* filter cloth composed of fine non-corrodible wire fabric, or a "filtros" plate, for uniformly distributing the flow of water or brine solution over and to the face of the base-exchange material.
W. T. LOCKETT.

Purifying waste waters. J. A. NEWLANDS (U.S.P. 1,557,188, 13.10.25. Appl., 19.1.21).—A process for the treatment of waste liquors containing sulphuric acid the object of which is twofold, viz., the prevention of the pollution of streams and the utilisation of the sulphuric acid in the production of lithopone. The waste water is treated with metallic zinc to produce zinc sulphate, the deposited copper and other impurities, *e.g.* iron, are removed, and barium sulphide is added to obtain a mixed precipitate of zinc sulphide and barium sulphate.
W. T. LOCKETT.

Material for combating pests. (G.P. 417,041).—See XVI.

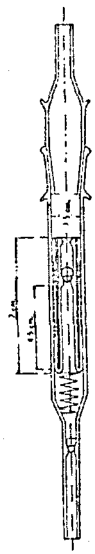
BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEBRUARY 19, 1926.

I.—GENERAL; PLANT; MACHINERY.

Laboratory pump for the circulation of gases. W. Francis (Fuel, 1926, 5, 39—40).—The glass piston (see figure) is moved up and down by a solenoid acting on a tube of soft iron enclosed within the piston. The solenoid is operated by a battery in series with a clockwork pendulum make and break. When the piston rises air is drawn through the lower glass valve, which closes while the piston falls, and the upper valve in the central tube of the piston opens and the gas flows through. A pump of the dimensions shown will give any rate of circulation up to 80 c.c. per min. A. B. Manning.



New methods of washing gases. V. Investigations with the aid of absorption curves. G. Weissenberger, R. Henke, and E. Sperling (Z. angew. Chem., 1925, 38, 1161—1164; cf. B., 1924, 773; 1925, 388, 697).—Absorption curves are obtained by plotting the degree of absorption (g. absorbed per 100 g. of absorbent) against the concentration of the gas phase (g. of vapour per cub. m. of air), the working temperature throughout being 20°. Decalin is slightly inferior to tetralin for absorbing benzene and alcohol, and much inferior for acetone and ethyl acetate. "Hydroterpin," a mixture of hydrogenated terpene hydrocarbons and tetralin, is practically non-absorbent for methyl alcohol, but absorbs ethyl alcohol, though less efficiently than tetralin, and accompanied by frothing; it is less efficient than tetralin for acetone and benzene, but is equal to tetralin, and better than turpentine or lignite tar oil, for absorbing lignite benzine, and has the advantage of imparting to the latter a pleasant odour. Absorption curves can be applied to the solution of a variety of problems, *e.g.*, determining the composition of a solvent without analysis (by comparison with absorption curves of known compounds), and the examination of the protective action of substances in admixture with solvents, *e.g.*, addition of tetralin to cresol for the absorption of the vapours produced in wood carbonisation, in which 60% of tetralin practically prevents thickening of the absorbent, whilst the efficiency of the latter only begins to be materially impaired with a content of upwards of 70% of tetralin.
W. T. K. Braunholtz.

Measuring dust in air and industrial gases. W. Allner (Z. angew. Chem., 1925, 38, 1170—1171).—Air or gas is drawn from the main through a sampling tube and passed through a cylindrical dust catcher which can be electrically heated and contains filter-cloth or the like, thence through an ice-cooled condenser and a calcium chloride tower (for water vapour), and finally through a gas meter. It is important that the gas pressure in the mouth of the sampling tube should be the same as that in the main, and this is regulated by means of a pump and a manometer, the limbs of which end respectively in the mouth of the sampling tube and in the main alongside. W. T. K. Braunholtz.

Active charcoals. Ruff.—See II.

Patents.

Apparatus for crushing ferruginous and agglutinative substances. A. Elenbaas (E.P. 236,578, 3.7.25. Conv., 4.7.24).—An apparatus for crushing material such as spent oxide from gas works comprises a hopper the bottom of which is formed by a roller having broaches on its surface. The opposite sides of the hopper have openings through which the broaches pass. S. Binning.

Gyratory [fine] crushing process and apparatus. E. Symons (E.P. 243,926, 2.2.25).*—The material to be crushed is fed between a pair of vertical conical crushing elements, both cones being inclined the same way, *i.e.*, the outer fixed cone or concave, envelopes the inner gyrating cone. The gyrating cone, C^1, is supported on a spherical bearing, A^9, the centre of which substantially coincides with the geometrical apex of the cone, so that the extent of the gyrating motion or crushing stroke is greatest at the skirt or lower edge of the cone and small near the top where the material enters, and the motion of any point on the cone is combined lateral and vertical. The gyratory motion is produced by the rapid rotation of an eccentric sleeve, B, around a shaft, C^7, extending downwards from the cone. The sequence of actions on any particle is as follows: first a drop by gravity from the feeding device, D^4 (Fig. 3), on to the gyratory cone at the top, where the crushing space is widest; an approach of the inner cone towards the concave, E, the motion being so rapid that the particle slides down the inner cone until it is nipped and crushed at the end of the approach stroke; the recession of the inner cone is then so

rapid that the crushed particles hang freely for a moment and drop vertically on to the inner cone at a point lower down than before, and on the re-approach

FIG. 2.

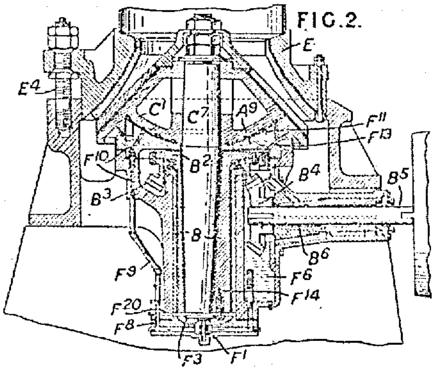
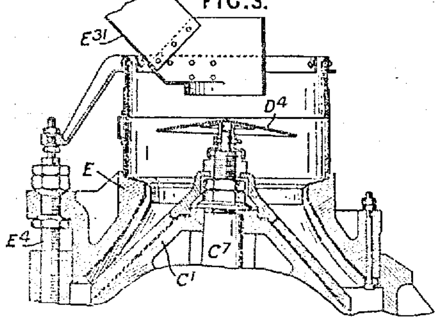


FIG. 3.



of the cone the particles continue their motion downwards by sliding until they are again nipped, and so on until they reach the skirt of the cone and are discharged. In order to effect fine crushing, to, say, ¼ in., with a large stroke at the skirt, say 2½ in. or certainly more than twice the size of the largest particles discharged, the surface of the concave or outer cone is not truly conical but is flared, so that at the upper part the crushing surfaces are convergent but in the lower part they are parallel for such a length that no particle has time to slide right through the parallel part without being nipped at least once, and hence the maximum size of the discharged particles will depend on the minimum gap between the crushing surfaces. For the correct functioning of this crusher it is essential that the material be not crowded, which requirement necessitates an automatic feed. This can be effected by means of a plate (conical, flat, or dished according to the angle of repose of the material to be crushed), D^4, attached to the top of the gyrating cone and working in conjunction with a fixed feeding hopper or guide which delivers the material to the centre of the plate. The adjustment of the size of the crushed product is effected by raising or lowering the outer concave by means of adjusting screws, E^4. Means are provided for forced lubrication and for preventing creep of the stationary concave. B. M. VENABLES.

Means of pulverising, and separating, all kinds of cereals, minerals, and the like. J. C. CARLINE (E.P. 244,146, 9.8.24).—The apparatus comprises two disintegrators and a spiral wire-brush conveyor through which the material passes in series, the conveyor delivering finished material while insufficiently crushed material is allowed to drop out of the circuit (before reaching the conveyor) and to return to the first disintegrator. The stationary grinding surface of the disintegrator is provided with adjustable spring-mounted or rigid blocks with which hinged extremities of the rigid arms of the rotor come in contact.

B. M. VENABLES.

Apparatus for separation of solid materials by means of liquid currents. H. REMY (E.P. 236,947, 8.7.25. Conv., 8.7.24).—The apparatus comprises a number of units in each of which a rising current of water separates and carries off the lighter constituents of a mixture of solids that is dropped into it, and the residual solid constituents forming the underflow are treated in a subsequent unit with a faster current of water. A transfer device is provided between the units which permits the residual solid matter to drop from one unit to the next without allowing any substantial intermingling of the currents of water in successive units, *i.e.*, it acts as a water-lock. The transfer device may consist of a cellular drum, an Archimedean screw, or of a number of flaps arranged in series and opened in succession. B. M. VENABLES.

Process and apparatus for precipitating and filtering. L. D. MILLS and T. B. CROWE (E.P. 242,383, 3.9.24).—A filtering apparatus, for example of the vacuum leaf type, suitable for the precipitation and collection of gold and silver from cyanide solutions, is arranged so that there is a brisk circulation of liquid or pulp over the surfaces of the filter medium, the filtrate being withdrawn comparatively slowly, but quickly enough to produce cakes which are uniform in texture and form an excellent final precipitating means because the coarse particles of sludge are not permitted to settle out. The circulation is preferably maintained by a propeller within a vertical shaft, placed centrally in the filter tank, the pulp passing up the shaft and being deflected downwards along the surfaces of the filter-leaves. B. M. VENABLES.

Filtering apparatus. W. HOEHN (U.S.P. 1,564,351, 8.12.25. Appl., 4.4.21).—In a filtering apparatus consisting of a number of filter-leaves in a tank, each of the filter-leaves with its respective holder is independently connected to a manifold, so that a filter-leaf with its holder can be removed from the tank without interrupting operation of the apparatus. S. BINNING.

Filter. F. B. LOMAX (U.S.P. 1,565,661, 15.12.25. Appl., 29.9.24).—A container has a horizontal filtering partition near the bottom and is provided with means for agitating the material above the filter and for withdrawing the filtrate. The whole is supported on a stand and the means for securing the apparatus to the stand includes the filtrate outlet. B. M. VENABLES.

Preventing adhesion of scale in boilers, heaters, evaporators, and the like. K. SCHNETZER (E.P. 243,415, 31.7.24. Addn. to 208,115; B., 1925, 231).—The method described in the original patent is modified by varying the direction of flow of the current at intervals, for example, by changing the points on the metallic surface to which the current is supplied. S. BINNING.

Separating oil from water contaminated with oil. E. W. GREEN, H. OGDEN, and G. R. UNTHANK (E.P. 243,501, 29.10.24).—Water contaminated with oil in a very finely dispersed condition is distributed over a large wetted area exposed to air, *e.g.*, a bed of coke. The oil is retained as a film, and gradually accumulates and is transformed into drops, which are removed by the flowing water and are recovered in a trap. S. BINNING.

Method of treating substances centrifugally and machines therefor. A. E. WHITE. From SHARPLES SPECIALTY CO. (E.P. 243,946, 24.2.25).—A centrifugal machine for separating two substances, at least one of which is liquid, by means of a third or carrier liquid, *e.g.*, paraffin wax from chilled oil with the aid of brine, is described. The 23 claims refer mainly to the various arrangements of the "weirs" or devices to regulate the separation and withdrawal of the products. B. M. VENABLES.

Means for emptying a revolving open-top centrifugal separator. E. VAN DER MOLEN (U.S.P. 1,565,604, 15.12.25. Appl., 26.9.24).—A scraping blade is drawn gradually into the collected material, which then passes along an inclined runway starting from near the blade and sloping gradually upwards over the opposite wall of the centrifugal drum. (Cf. E.P. 238,460; B., 1925, 790.)

B. M. VENABLES.

Evaporating solutions *in vacuo*. N. V. NEDERLANDSCHE INSTALLATIE MAATSCHAPPIJ THERMA, and A. O. H. PETERSEN (E.P. 243,982, 1.5.25).—The vapours from a hot solution which has been evaporated *in vacuo* are condensed in a condenser by just sufficient cooling liquid to produce condensation, and the condensate is removed by an ejector pump actuated by the cooling liquid used in the condenser. The method is particularly suitable for the recovery of Epsom salts, Glauber salts, and potassium salts from their solutions.

S. BINNING.

Vacuum drying [evaporating] apparatus. H. D. MILES, Assr. to BUFFALO FOUNDRY AND MACHINE CO. (U.S.P. 1,560,598, 10.11.25. Appl., 31.12.24).—An internally heated drum is rotated inside a chamber maintained under reduced pressure by a suction device. The liquid under treatment is delivered to the bottom of the closed chamber and is rapidly evaporated in the form of a film on the surface of the drum. The dried solid deposited is removed by a scraper from the surface of the drum before it again enters the liquid. The vapours exhausted from the chamber are drawn through the liquid to be evaporated so that any solid entrained in them is recovered. The apparatus is particularly useful in the preparation of milk powder.

T. S. WHEELER.

Apparatus for separating moisture from gases in vacuum drying plants. GEN. ENGINEERING CO. (RADCLIFFE), LTD., and S. TAYLOR (E.P. 244,233, 1.11.24).—A condenser for condensing and rendering visible the moisture in the gases leaving a vacuum drying oven is constructed of a number of groups of water-cooled tubes giving alternate upward and downward flow of the gases. The total cross-sectional area of the tubes increases in successive groups so that as the gases cool they pass through the tubes at a lower velocity. The condensed water is collected where the gases change direction at the bottom of one set of tubes in order to pass up the next, and arrangements are made for drawing off the condensed water without breaking the vacuum.

B. M. VENABLES.

Method of drying and oxidising materials in suspended condition. Drying materials. G. D. HARRIS, Assr. to INDUSTRIAL DRYER CORP. (U.S.P. 1,564,565–6, 8.12.25. Appl., [A] 23,10.20, [B] 18.2.24). —(A) Suspended material is moved within a chamber from which a definite volume of the drying atmosphere is removed in a damp condition and divided into a number of streams, each of which is separately conditioned by mixing with fresh air and by reheating, and then again circulated within the drying chamber. (B) In a drying apparatus where the atmosphere is reheated while circulating and some of the atmosphere is withdrawn, conditioned by exchange with fresh air, and again circulated, the periods of reheating are controlled by the changes of temperature of the atmosphere and the periods of conditioning by the moisture content of the atmosphere.

B. M. VENABLES.

Method and apparatus for drying. Method and apparatus for drying, including solvent recovery. G. D. HARRIS, Assr. to INDUSTRIAL DRYER CORP. (U.S.P. 1,564,782–3, 8.12.25. Appl., 6.5.21).—(A) An atmosphere circulating within a drying chamber is expanded by heat and the excess permitted to escape, thus producing a rarefied drying atmosphere, which is then alternately cooled and heated to set up a circulation while excluding external air. (B) A drying chamber with an attenuated circulating atmosphere is provided with a number of internal flues, leading from the top of the chamber to the bottom, through which the circulating atmosphere moves downwards in separate paths. Condensers are provided in each flue near the top and heating means near where the flues open into the main chamber (at the bottom) and "boosters" in the chamber at points between the bottom and top. B. M. VENABLES.

Heating system for kilns. J. B. AGNEW and A. A. OEFFNER (U.S.P. 1,562,135, 17.11.25. Appl., 12.3.25).—Air or oxygen under pressure is introduced into the top of a down-draught kiln and meets the hot gases rising from the fire-boxes,

producing a whirling motion of the products of combustion which ensures thorough heating of the material in the kiln. T. S. WHEELER.

Furnace. N. I. IVANOVSKY (U.S.P. 1,565,370, 15.12.25. Appl., 27.12.24).—A furnace comprises a combustion chamber, a regenerator, and a gas producer, the last being adjacent to, and provided with apertures communicating with, the other two. A number of brick-walled passages in the regenerator and also the walls of the gas producer are heated by the exhaust gases, and the air for combustion is admitted partly through the regenerator and partly through the gas producer. B. M. VENABLES.

Digestion and filtration apparatus. L. C. DANIELS, Assr. to NAT. ANILINE AND CHEMICAL Co. (U.S.P. 1,562,870, 24.11.25. Appl., 9.1.20).—A combined extraction and filtration apparatus consists of a steam-jacketed vessel connected with a reflux condenser and having an outlet pipe which passes to the bottom of the apparatus and is fitted with a filter head. The material under treatment is heated with the solvent and when extraction is complete it is blown out through the filter head by means of compressed air. T. S. WHEELER.

Apparatus for refrigeration. B. B. HOLMES (U.S.P. 1,564,242, 8.12.25. Appl., 13.2.22).—A refrigerating machine consists of a boiler and condenser with an intermediate ejector connected with an evaporator. Boiler, condenser, and evaporator contain a volatile liquid the vapour of which is drawn regularly from the evaporator and discharged into the condenser. Means are provided for periodically stopping the action of the ejector, and for supplying the liquid to the discharge end of the ejector when the jet is not acting, for return to the evaporator. A. COUSEN.

Dephlegmator. J. W. HANCOCK (U.S.P. 1,564,564, 8.12.25. Appl., 16.2.23).—A tower is provided with a central vertical flue, the surfaces of both the flue and the tower being air-cooled. In the annular space within the tower are a number of annular collecting chambers or deep trays, the walls of which are spaced from the central flue and from the walls of the tower. B. M. VENABLES.

Apparatus for absorption of gases in liquids. H. HOWARD, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,565,366, 15.12.25. Appl., 6.12.22).—A closed chamber contains the liquid which is to be impregnated with gas and is provided with a horizontal plate with many small perforations, to the underside of which the gas is supplied. The plate has a central large aperture in which is situated a device, such as a propeller, for circulating the liquid and causing it to pass radially over the plate. For use in the manufacture of sulphuric acid the plate may be of cast iron and divided into separately detachable sections. B. M. VENABLES.

Production of membranes of parchment paper for osmotic purposes. SIEMENS U. HALSKE A.-G. (G.P. 418,210, 19.9.22).—Membranes for the dialysis of soap solutions etc. are produced by treating parchment paper with caustic soda or potash. D. F. TWISS.

Rotary drying apparatus. P. SCRIVE (U.S.P. 1,567,335, 29.12.25. Appl., 12.1.24).—See E.P. 224,111; B., 1925, 56.

Absorption refrigerating machines. A. S. GRAY (E.P. 230,079, 26.2.25. Conv., 29.2.24).

Refrigerating apparatus. A. GREEN (E.P. 244,532, 22.9.24).

Rabbling apparatus. EBERHARD HOESCH UND SÖHNE (E.P. 237,251, 13.7.25. Conv., 21.7.24).

Hydrometer. F. C. and H. S. JEWELL (E.P. 244,669, 20.7.25).

Optical pyrometers. SIEMENS U. HALSKE A.-G. (E.P. 244,691, 16.10.25. Conv., 29.5.25.)

Rotary drum apparatus for drying, heating, and mixing granular and other [road-making] materials. J. M. T. JOHNSTON and J. E. DAVIES (E.P. 244,975, 1.4.25).

Apparatus for draining water from steam-heated rotary cylinders and the like. J. M. ARNOT (E.P. 245,031, 30.7.25. Addn. to 239,087).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Limits of inflammability of firedamp and air. M. J. BURGESS and R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 15, 1925. 21 pp.).—The effect of varying experimental conditions on the limits of inflammability of mixtures of methane and air has been studied. The dimensions and material of the containing vessel have no appreciable effect on the limits of inflammability when determined in tubes of diameter greater than 5 cm. The values for the horizontal propagation of flame in wider tubes, open at one end, are 5·4% CH_4 and 14·3% CH_4. For tubes of less than 5 cm. in diameter, the lower limit increases and the upper limit decreases with decreasing diameter until a minimum diameter is reached (4·5 mm. for glass tubes); in narrower tubes no mixture of methane and air is capable of continued propagation of flame. The limits for upward, horizontal, and downward flame propagation are 5·4 and 14·8%, 5·4 and 14·3%, and 6·0 and 13·4% respectively. For horizontal propagation of flame there is little difference in the limits when the mixture is totally confined and when it is free to expand. For upward propagation, however, the lower limit is least when the mixture is free to expand and the upper limit is greatest when the mixture is totally enclosed. The limits widen with rise of temperature, and with increasing pressure from 120 mm. to about 760 mm., when the lower limit passes through a minimum. This variation with pressure is observed with other gases of the paraffin series, but the position of the lower limit

minimum varies, being, for example, about 4 atm. for pentane. Such variations in temperature and pressure as ordinarily occur in coal mines do not appreciably affect the limits of inflammability. A mixture of methane and air containing about 5% CH_4 can propagate flame (*a*) under certain limiting conditions of turbulence of the mixture, or (*b*) when the mixture is travelling as a slow current. The presence of water vapour in the atmosphere does not appreciably affect the lower limit of inflammability. The reduction of the oxygen content of the air narrows the limits, the upper limit being affected more than the lower, until, when the air contains 13% O_2, they coincide and only one mixture, containing about 6% CH_4, can propagate flame. If the reduction of the oxygen content is brought about by the addition of carbon dioxide, the limits are narrowed more rapidly, owing to the specific heat of carbon dioxide being higher than that of nitrogen. The effect of the presence of another combustible gas is dependent on the nature of that gas and can be calculated from the known values of its limits of inflammability with air alone. A. B. MANNING.

Determination of carbon in coal. J. G. KING and D. MACDOUGALL (Fuel, 1926, 5, 33—35).—The carbon in a strongly caking bituminous coal, high in volatile matter (Wearmouth gas coal), was determined by the "combustion" method in an electrically-heated furnace of standard pattern (*cf.* Fuel Research Board Interim Report on Methods of Analysis of Coal). The temperature of the copper oxide was varied in stages from 800° down to 350°, and the amount of carbon escaping combustion was determined by measurement and analysis of the gases passing through the absorption vessels. The standard method of combustion of the Sampling and Analysis of Coal Committee (*loc. cit.*) has been shown to give satisfactory results. The time taken to burn the coal should not be less than 30 min., and the total time of one determination need not exceed 120 min. The temperature at the centre of the layer of copper oxide should not be lower than 800°, and at this temperature, even with rapid heating, there is no likelihood of the loss of carbon exceeding 0·15%. At temperatures lower than 800° the amounts of carbon escaping combustion increase. At 700° and at 650° results to within 0·3% can be obtained by careful manipulation, but variations of over 1% tend to occur. A. B. MANNING.

Fusibility of coal ash. A. C. FIELDNER and W. A. SELVIG (Fuel, 1926, 5, 24—33; cf. Bull. 209, U.S. Bureau of Mines, 1922; B., 1922, 738 A).—The requirements of a standard method for the determination of the fusibility of coal ash have been studied. In particular, the ash should be heated in an atmosphere which reduces the iron component to the ferrous state, in which it exerts its maximum fluxing action. The reducing atmosphere of a gas-fired furnace fulfils this requirement. The standard method of the U.S. Bureau of Mines is described in detail, and the results of determinations on a large number of American coals are summarised. A. B. MANNING.

Bergius process for the liquefaction of coal. F. SCHUSTER (Sparwirtsch., 1925, 3, 157).—The apparatus, procedure, and the products obtained by the Bergius process (G.P. 301,231 and 303,893; cf. E.P. 18,232 of 1914 and 5021 of 1915; B., 1916, 167, 732) are briefly described. The process is illustrated by the following results of a large-scale experiment: 1 ton of dry coal, with an ash content of 4%, yielded 210 kg. of gas, 455 kg. of oil, 75 kg. of water, and 5 kg. of ammonia. The residue was carbonised, and gave further 25 kg. of gas, 80 kg. of oil, and 240 kg. of coke. In all, 535 kg. of oil were obtained, which on fractionation yielded 150 kg. of refined benzine, 200 kg. of Diesel oil, and 60 kg. of lubricating oil; the residue served as fuel oil. The Bergius plant at Mannheim-Rheinau has a reaction cylinder of 4 m.^{3} capacity, and can deal with 18,000 tons of raw material per annum. A. B. MANNING.

Active charcoals and their adsorptive power. O. RUFF (Z. angew. Chem., 1925, 38, 1164—1169; cf. B., 1924, 362; 1925, 117).—Activated and non-activated charcoals may be distinguished by their percentage adsorptive power for phenol, *i.e.*, one hundred times the quantity of phenol adsorbed by 1 g. of charcoal from 100 c.c. of a 1% phenol solution at 25°. The differences in adsorptive power towards gases and dissolved substances suggest two different types of attraction. Elementary analysis affords no clue as to activity, but active charcoals are always denser than inactive charcoals. For charcoal to be capable of activation it must be amorphous, activation being effected by any means (*e.g.*, heating in air, steam, carbon dioxide) that causes the amorphous surface to be etched or partially broken up. The loss of carbon in the process need not be great (*e.g.*, 6½%). Carbonisation of various organic substances out of contact with air and in the presence of certain inorganic compounds (*e.g.*, alkalis) leads direct to active charcoals. Heating to too high a temperature (*e.g.*, 1100° and above) destroys the activity and capacity for activation of amorphous carbon. Active amorphous carbon probably contains irregular groups of carbon atoms with valencies incompletely satisfied, and where these lie on or near the surface or the pores of the charcoal they act as centres of adsorptive attraction. In inactive charcoals the amorphous substratum is covered with a dense skin of saturated carbon atoms, and the object of activation is to remove or break through this skin. W. T. K. BRAUNHOLTZ.

Analysis of organic substances of high ash content, in particular of Esthonian shale. E. VON PEZOLD (Brennstoff-Chem., 1925, 6, 381—385).—The chemical composition of the inorganic constituents present in the material should be determined before deciding upon the best method of determining the ash content. Direct incineration, as usually practised,

will give erroneous results if the inorganic matter comprises oxides (which will take up sulphur dioxide from gas), hydrates (which will give up water at elevated temperatures), and carbonates. In such cases, the dried and weighed material should be first treated with 10% hydrochloric acid, the residue filtered off, dried, and weighed, and finally incinerated in the usual manner. The loss in weight on incineration of the residue from the acid extraction gives the content of organic matter, and from this the ash content of the original sample is calculated. This method must, however, be used with caution for coals and other easily oxidisable substances, owing to the possibility of oxidation occurring in the drying oven and thus leading to unduly high values being obtained for the organic matter.

W. T. K. Braunholtz.

Development of a standard Canadian laboratory distillation method for examination of oil shale. R. E. Gilmore and A. A. Swinnerton (Canad. Chem. Met., 1925, **9**, 215—217, 235—239).—A standard shale sample with an oil content of about 30 gals. per short ton was retorted by various current methods. These comprised a cross retort of 3500 g. capacity immersed in a lead bath electrically heated, 3000 g. of shale being used and the temperature raised to 550°; the small field assay retort of the U.S. Bur. of Mines, charged with about 350 g. of shale and heated in an electric furnace; a large field assay retort, charged with 1000 g. of shale, heated by a gas burner or in an electric furnace; a horizontal iron tube, 2½ in. inside diam. and 26 in. long, heated in a gas furnace and charged with 450 g. of shale, and the Scotch (Pumpherston) tube method with an iron tube, 2 in. in diam. and 6 ft. long, closed at one end and open at the other, which is charged with 450 g. of shale and heated in an inclined position, so that the distillate runs out of the open end. Distillations of the crude oil were made in an Engler flask on 100 c.c. and in a Hempel flask on 300 c.c. The lead bath retort was found inconvenient. The assay retorts gave the most accurate results, the larger one being preferred. The Scotch tube method gave only 29 gals. of oil per ton, as against 32 gals. by the small assay retort and about 31 gals. by the large assay retort. The yield of gas without use of steam was 1095—1635 cub. ft. per ton, and with the use of steam up to 4020 cub. ft. per ton. The crude oil from the large assay retort had *d* 0·881, and on distillation yielded 8% up to 150°, 31% from 150 to 300°, 46% from 300° to end point, and 15% of pitch and loss.

H. Moore.

Thermal expansion of California petroleum oils. E. H. Zeitfuchs (Ind. Eng. Chem., 1925, **17**, 1280—1283).—Various Californian petroleum products were tested to ascertain their coefficients of expansion from 50° up to 400°. Glass pycnometers were used up to 200°, and steel pycnometers for higher temperatures. These were heated in vapour from baths of liquids of suitable boiling points or in baths of melted solder. A single formula could not be found to cover the expansion over the whole range. The coefficient increased rapidly at higher temperatures, and was greater for high temperatures than with oils of other fields. A graph indicates the error introduced by using the National Standard Petroleum Tables for computing the gravities of these products; this error is 7% for 50° A.P.I. gravity.

H. Moore.

Sulphur compounds removed from a Persian petroleum by means of sulphuric acid. I. E. H. Thierry (J.C.S., 1925, **127**, 2756—2759).—The oil obtained on dilution of a sulphuric acid sludge, from the refining of Persian petroleum, has been separated into 29 fractions boiling below 120°/125 mm., together with a residue. Fraction (4), b.p. 68—70°/760 mm., contained methyl ethyl sulphide, which was characterised by preparation of its mercuri-iodide, the methylsulphonium iodide mercuri-iodide, and *methyl ethyl sulphide mercurichloride*, m.p. 101—102°. Fraction (7), b.p. 89—92°/760 mm., contained a compound $C_4H_{10}S$, of which the mercuri-iodide, unmelted below 100°, methylsulphonium iodide mericuri-iodide, m.p. 54°, and mercurichloride, m.p. 68°, are described; this compound is not ethyl sulphide. Fraction (11) contained a liquid, b.p. 64—65°/125 mm., 120—121°/760 mm., which was recognised as tetramethylene sulphide; the *methiodide*, decomp. 185—190°, mercurichloride, m.p. 126°, *tetramethylene sulphide mercuri-iodide*, m.p. 58°, and *tetramethylene methylsulphonium iodide mercuri-iodide*, m.p. 111°, are described. Fraction (17) contained pentamethylene sulphide, b.p. 83—84°/125 mm., 138·5°/742 mm., which was oxidised to pentamethylene sulphone, m.p. 98°; the *sulphonium chloride*, decomp. on heating, was prepared from the sulphonium iodide through the sulphonium base; *pentamethylene sulphide mercuri-iodide*, m.p. 72—74°, the mercurichloride, m.p. 135—136°, the *chloroplatinate*, and *pentamethylemethylsulphonium iodide mercuri-iodide*, m.p. 78°, are described.

F. M. Hamer.

Sulphuric acid absorption and iodine values of various petroleum products and cracked distillates. J. C. Morrell and G. Egloff (Ind. Eng. Chem., 1925, **17**, 1259—1261).—The amounts of various oils dissolved by sulphuric acid, *d* 1·84, are disproportionate to their content of unsaturated substances, 100% of some crude oils being absorbed. For determining iodine values the method of Hanus was used on a solution of oil in chloroform containing 0·01 g. per c.c. 25 c.c. of Hanus solution were used and an absorption period of 30 min. in the dark. When the proportion of reagent to oil solution was increased the iodine value greatly increased. There was considerable change in the addition iodine value with changed proportions of the reactants, and a still greater change in the substitution value.

H. Moore.

Theory of anti-detonators. H. Muraour (Chim. et Ind., 1925, **14**, 851).—The author suggests that the action of anti-detonators, such as lead tetraethyl in internal-combustion engines, is due to the formation of a cloud of solid particles, which

fix a proportion of the ions in the gas, and thus moderate the rate of explosion. The fact that benzene–air mixtures can be strongly compressed without detonation is ascribed to initial combustion of the hydrogen, whereby a cloud of carbon particles is formed and acts as an anti-detonator.

L. A. COLES.

Catalytic formation of petroleum hydrocarbons from fats. J. MARCUSSON and W. BAUERSCHÄFER (Chem.-Ztg., 1925, **49**, 1045).—Conversion of fats into petroleum hydrocarbons takes place at lower temperatures than those used by Mailhe (cf. B., 1921, 650 A, 803 A), the low-boiling hydrocarbons obtained by Mailhe being secondary decomposition products of heavier oils, which are the primary products of the transformation. Mixtures of oils or fats with one-fifth of their weight of uncalcined kieselguhr or sodium chloride were boiled for 20 hrs. under a reflux condenser, the temperature being kept below 300°. After removing saponifiable constituents by treatment with alkalis, the products from linseed oil and tallow were a moderately viscous brown liquid with green fluorescence and a brown liquid of low viscosity, having d^{20} 0·9219 and 0·8762, iodine value 52 and 29·3, acetyl value 13·6 and 16·6, and acetyl value after hydrogenation 31·8 and 61·3, respectively. In each case the product consisted mainly of saturated and unsaturated hydrocarbons, and contained alcohols and ketones in addition. The product from tallow, on fractional distillation yielded 18% of b.p. 150—300°, and a viscous residue, d 0·91. L. A. COLES.

Methods of testing transformer oils. H. C. STAEGER (Ind. Eng. Chem., 1925, **17**, 1272—1275; cf. B., 1924, 898; 1925, 531).—The author reviews the composition and qualities of transformer oils and methods of test as given by different authorities. Graphs illustrate the oxidation and sludging of oils over prolonged periods in the presence of various metals. The temperature of the Michie test (150°) is considered too high, and the best agreement with the results of actual service is obtained with the B.B.C. test, in which the oil is heated to 112° in a copper beaker for 300 hrs., and the sludge determined gravimetrically. H. MOORE.

Methods of washing gases. WEISSENBERGER, HENKE, and SPERLING.—See I.

PATENTS.

Fuel dryer. H. KREISINGER, Assr. to COMBUSTION ENGINEERING CORP. (U.S.P. 1,564,361, 8.12.25. Appl., 3.10.24).—Within a casing is a heated conduit through which the fuel is passed. The conduit divides the casing into two chambers, one of which is provided with a gas inlet, and the other with a gas outlet, so that a current of gas can be passed through the fuel as it is being dried. A. B. MANNING.

Pulverising, feeding, and burning fuel. RAYMOND BROS. IMPACT PULVERIZER CO., Assees. of J. CRITES (E.P. 230,405, 29.5.24. Conv., 8.3.24).—In a system for pulverising fuel and feeding it to a furnace in a current of air, the fineness of pulverisation being controlled by an adjustable device (cf. E.P. 101,962 or 103,963) situated between the pulverising chamber and the fan chamber, a controlled volume of air is admitted to the pulverising chamber, and an independently controlled volume of additional air is admitted to the fan chamber. When so using the adjustable device referred to, it is possible to carry the pulverised fuel from the fan chamber to the furnace in a current of air at a high pressure, notwithstanding that the pressure of the air carrying the material through the pulverising chamber is maintained relatively low. A. B. MANNING.

Burning pulverised fuel. L. H. BERGMAN (E.P. 232,268, 14.4.25. Conv., 14.4.24).—Pulverised fuel is conveyed from the pulverising mill to a storage bin in a current of hot gases circulating through the apparatus, and passes from the bin to the furnace before it has had time to cool. The hot gases may be flue gases derived from the furnace. Provision is made for adding fresh hot gases as required from the furnace to the circulating system, and at the same time withdrawing a portion in order to prevent undue accumulation of moisture in the gas stream. The fuel is partially dried by this treatment, and clogging of the feeding mechanism due to condensation of moisture in the storage bin is prevented.

A. B. MANNING.

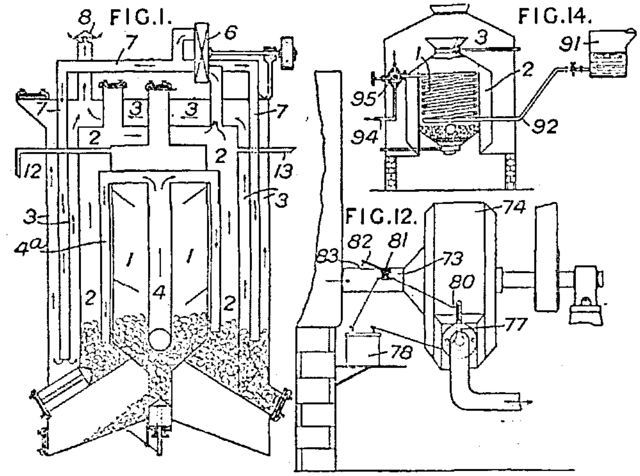
Briquette. A. D. PARKER, Assr. to AMER. BRIQUET CO. (U.S.P. 1,564,890, 8.12.25. Appl., 8.1.25).—Starch is mixed with borax in water, a relatively large quantity of water added thereto, and the whole heated to boiling point. Hot asphaltum is added and stirred in to form an emulsion, which is then mixed with culm or the like, and the mixture formed into briquettes and dried.

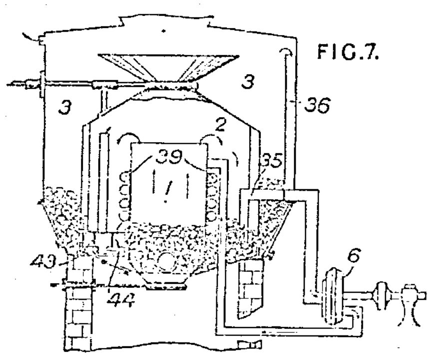
A. B. MANNING.

Coking coal. P. C. RUSHEN. From KOPPERS CO. (E.P. 243,414, 30.7.24).—The oven tapers from top to bottom, and steam inlets are provided in the floor of the carbonising chamber. The lower portion of the charge receives the first impact of the coking heat, and when this portion is completely coked, steam is admitted to produce blue water-gas, the steaming being continued until the remaining thicker portion of the charge is completely coked. The vertical flues of the heating walls on opposite sides of the coking chamber may be connected by cross-over ducts. A. R. MYHILL.

Carbonising fuels. F. KRAUSS (E.P. 243,534, 8.12.24).*—An apparatus for carbonising or gasifying fuel, particularly moist fuel, *e.g.*, peat, lignite, etc., comprises an inner chamber, 1, to which either hot furnace gases are supplied through a tube, 4, or in which the fuel is burned in an air supply, surrounded by an annular carbonising chamber, 2, which in turn is surrounded by a fuel drying chamber, 3. Hot gases from the chamber pass through branches, 4*a*, from the pipe, 4, into contact with the fuel in chamber 2, the gases from which are circulated by a fan, 6, and tubes, 7, through the fuel in the drying chamber, 3. The

cooled gases escape through the tube, 8. Combustible gases are led off through the tubes, 12, 13, for use in the chamber, 1, or elsewhere. Fuel is charged into,

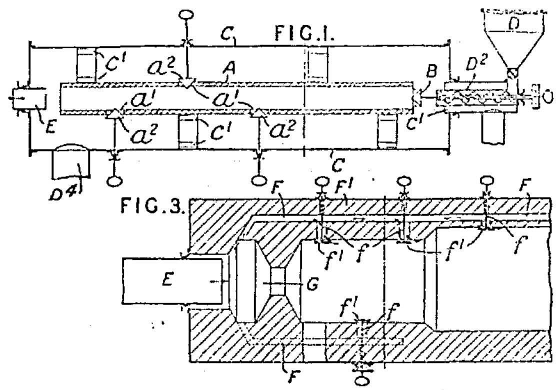




and coke discharged from each chamber separately. In a modification shown in Fig. 7, the hot gases are withdrawn by tubes, 35, 36, and fan, 6, and are heated in a coiled pipe, 39, leading, according to the position of a valve, 43, to chamber, 2 or 3, or when a valve, 44, is open, to chamber, 1. The fan, 74 (Fig. 12), withdrawing the gases may be controlled automatically by the heat of these gases, by providing in the outlet pipe, 73, a valve, 82, operated by a solenoid, 81, the circuit of which and a battery, 78, is closed when the contact element, 80, in a thermometer, 77, is reached, the valve being opened and air admitted through an opening, 83, thus reducing the suction effect of the fan and cooling the outgoing gases. Liquid such as water or hydrocarbons from a tank, 91 (Fig. 14), may be vaporised in a coil, 92, in chamber, 1, and steam or oil vapour supplied through a valve, 95, either to the carbonising or drying chamber, or to a pipe, 94, leading to a motor.

A. R. MYHILL.

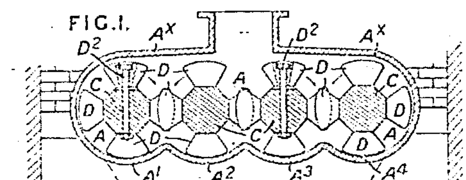
Distillation of carbonaceous materials. B. LAING and H. NIELSEN (E.P. 243,857, 10.10.24).*—In the destructive distillation of carbonaceous material at low or medium temperatures means are provided for admitting the heating gas to, and for allowing it to escape from, certain predetermined zones of the retort. For example, the hot gases enter the retort, C (Fig. 1), through the inlet, A^2, and a portion enters the concentric tube, A, and flows through openings, a, controlled by valves, a^2, into contact with the material, which is fed in through a conduit, D^2, and discharged through an opening, D^4. The mixed heating gases and distillates leave by a conduit, C^1. The tube, A, is movable longitudinally in the retort. In a modification



(Fig. 3) ducts, F, are formed in the retort lining, F^1, and the heating gases, throttled by a constriction, G, are forced along the ducts and through the openings, f, controlled by valves, f^1. The retort may be inclined or vertical, or may be rotary, and when treating strongly caking coals these may be passed through a heating and a cooling zone before being further heated, thereby avoiding clogging of the retort.

A. R. MYHILL.

Retorts for destructive distillation or heat treatment of solid materials. A. L. J. QUENEAU (E.P. 243,942, 23.2.25).*—Apparatus for the distillation or heat treatment of solid material comprises three or more horizontal retort units, A, in parallel



juxtaposition and in open communication with one another along their length. A separate conveyor, C, carrying blades or knives, D, runs through each retort. The blades revolve in such a manner as to intersect with and pass between those of the adjacent retort or retorts, without touching. Each conveyor shaft revolves in the opposite direction to the adjacent one. The bottom of the apparatus comprises a set of curved channels, A^1 to A^4, extending from end to end of the retorts. The material is charged at the top at one end of the retort and discharged at the bottom at the other end.

A. R. MYHILL.

Producing coke, gas, and tar from solid fuel. J. RUDE (E.P. 244,337, 11.5.25; cf. E.P. 204,718 and 228,763; B., 1924, 663; 1925, 275).—Solid fuel is carbonised in a two-stage process, passing first through an internally heated preheater in which its temperature is raised by a hot gaseous medium to a point below that at which the plastic stage commences. This medium may be air or fuel gas (or, in some instances, fuel gas diluted with flue gases), and is afterwards utilised, together with the volatile matter obtained from the preheating stage, in the combustion chambers of an externally heated retort, in which the carbonisation is completed. The gaseous medium used for preheating is itself heated by the retort flue gases in a heat interchanger, or, in a second modification, by the sensible heat of the produced coke. The process is applicable to both low-temperature and high-temperature carbonisation plants. A. B. MANNING.

Activated carbon for medicinal purposes. J. N. A. SAUER (E.P. 243,801, 4.9.24. Addn. to 174,702; cf. B., 1922, 232 A).—Activated carbon of high adsorptive power is prepared from vegetable material by carbonising the latter (*e.g.*, up to 800°), certain gases (*e.g.*, hydrogen, producer gas, steam) being preferably passed through the material during carbonisation. The product is activated in a state of suspension or movement by means of a gas (*e.g.*, air, superheated steam, chlorine) at an elevated temperature (*e.g.*, 400—1200°). It is finally purified by treatment with acid and alkaline solutions, as specified in the parent patent. W. T. K. BRAUNHOLTZ.

Removing chemicals from solids and semi-solids. [Distilling wood.] A. F. FRAME (U.S.P. 1,562,880, 24.11.25. Appl., 14.11.21).—An apparatus for distilling wood consists of an externally heated inclined cylinder. The wood under treatment is fed into the upper end of the cylinder and is carried through it by a screw conveyor. Means are provided for feeding the wood to the cylinder without admitting air or allowing distillation products to escape. T. S. WHEELER.

Oil-gas apparatus. L. BLANCHET, Assr. to SOC. DE CHIMIE ET DE LA CATALYSE IND. SIEGE SOCIAL (U.S.P. 1,565,409, 15.12.25. Appl., 1.8.21).—An oil-gas retort comprises an upper vaporising compartment, which communicates directly with a lower catalysing compartment. Means are provided for supplying a liquid hydrocarbon to the upper compartment, the vapour then passing over catalysts disposed in rows upon the floor of the lower compartment. A. B. MANNING.

Gaseous fuel. J. HARRIS (U.S.P. 1,565,933–5, 15.12.25. Appl., [A, B], 6.12.23, [C] 16.2.24).—A cutting gas is prepared by mixing (A) natural gas and ether vapour, the latter constituting not less than 2½% by vol. of the mixture, (B) carbon monoxide and ether vapour, the latter constituting not less than 2½% by vol. of the mixture, or (C) casing-head gas and ether vapour, the latter constituting approximately 5% by vol. of the mixture. A. B. MANNING.

Porous mass [for absorbing acetylene]. A. B. RAY, Assr. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,565,328, 15.12.25. Appl., 28.10.21).—A porous filling material for acetylene storage tanks is prepared by bonding fragmentary activated carbon with a bonding agent not substantially impairing its absorptive capacity. A. B. MANNING.

Distilling oil shale, bituminous coal, and the like to obtain light hydrocarbons. A. L. MOND. From AMERICAN SHALE REDUCTION CO. (E.P. 244,275, 22.12.24).—Carbonaceous material is heated in a rotary retort comprising two chambers, a preheating chamber in which it is raised to a temperature at which the volatile matter is about to be driven off, and a second chamber in which it is subjected, in thinner layers, to a higher temperature. Means are provided, *e.g.*, a bath of molten metal, for applying heat evenly to the second chamber. The volatile matter is released quickly, and is withdrawn by suction. No steam is introduced into the retort, but hydrogenation of the unsaturated volatiles produced by cracking is brought about by the hydrogen liberated by the decomposition of the moisture originally in the material. This reaction is catalysed by causing the vapours to pass through a cascade of hot spent shale in the second chamber. The vapours are condensed in stages, the first condenser removing the heavy paraffin wax fraction, the second the lubricating oil fraction, and the third the gasoline fraction. The first condenser is maintained at a temperature at which some of the heavy condensates are cracked, and the second at a temperature at which some of the remaining condensates are cracked, the temperatures being maintained by hot flue gases from suitably heated zones of the combustion chamber. A. B. MANNING.

Oil shale retort. S. H. CORFIELD (U.S.P. 1,562,541, 24.11.25. Appl., 10.5.22. Renewed 26.8.25).—The shale is passed through a casing closed at the top and bottom. The casing contains a number of superimposed tables, and baffle-plates direct heat around the tables. Scrapers above each table are carried by a frame, and blades cut in them are staggered in relation to the uncut portions. The rotation of the scrapers moves shale from one table to the one below through openings in the tables offset from each other. H. MOORE.

Recovering oil from shale. C. S. FOGH, Assr. to ORE ROASTING DEVELOPMENT CO. (U.S.P. 1,563,271, 24.11.25. Appl., 26.11.20).—The volatile constituents of oil shales and oil sands are vaporised in a continuous process by heating the material during its passage through a gas-tight, mechanically-rabbled furnace by heated non-oxidising gases. The gases are continuously removed from the furnace

and their volatile constituents condensed. The fixed gases are reheated and again passed into the furnace. H. MOORE.

Oil cracking stills. W. J. MELLERSH-JACKSON, From SINCLAIR REFINING Co. (E.P. 240,355, 16.2.25).—In oil cracking stills, to avoid excessive heating of the parts containing the oil, and at the same time to maintain a high temperature in the furnace and promote economic combustion of the fuel, preheated air is introduced into the furnace, and the furnace gases before they come into contact with the parts containing the oil are cooled to the desired extent by addition of flue gases withdrawn from near the outlet of the heating chamber. The flue gases may be mixed with the products of combustion in the fire-box, or between the fire-box and the heating chamber. Gases issuing from the heating chamber are used to preheat air in a vertical tubular preheater, and this air is introduced into the fire-box. The amount of flue-gases returned may be regulated by a steam jet, and the gases may be introduced around the walls of the fire-box, so as to protect parts of the furnace from the heat of combustion. The pressure employed in cracking various products may vary from 90 to 700 lb./in.^{2}, and the charging stock may pass once or more than once through the tubes. The oil in the tubes passes in counter-current to the heating gases. H. MOORE.

Process of making motor spirits and refining petroleum products. B. T. BROOKS, Assr. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,563,012, 24.11.25. Appl., 1.5.18).—A petroleum product is cracked under conditions giving a product rich in olefines. This product is subjected to the action of sulphuric acid of less than 66° B. (*d* 1·84). The oil is then separated from immiscible substances, treated with alkali, and distilled. H. MOORE.

Oil-cracking and carbon-removing method and apparatus. L. D. WYANT (U.S.P. 1,563,818, 1.12.25. Appl., 20.3.22).—Oil is heated to cracking temperature and agitated to maintain the liberated carbon in suspension. Portions of oil containing charges of carbon are ejected from the body of heated oil by the gases evolved from the oil. These charges are separated from the oil while within the still, and discharged. The still is provided with a collector above the level of the oil, and with conductors dipping into the oil to collect evolved gases and convey oil and carbon carried by the gases into the collector, where means are provided for separating the carbon from the oil and discharging it. H. MOORE.

Recovery of gasoline from natural gas. H. A. MOSSOR, Assr. to SOUTH PENN OIL Co. (U.S.P. 1,565,749, 15.12.25. Appl., 12.12.22).—Gasoline is recovered from natural gas by successively cooling the gas under compression, and compressing it in the presence of a solvent for the residual gasoline, the increase in density of the solvent, due to the absorption of constituents of the gas, not being allowed to exceed 1°B. After removing the residual gas, the pressure on the solvent is reduced so as to vaporise the dissolved gasoline, and the vapour is added to further supplies of the gas. L. A. COLES.

Manufacturing lubricating oils. R. W. HANNA, O. E. CUSHMAN, and T. W. DOELL, Assrs. to STANDARD OIL Co. (U.S.P. 1,566,000, 15.12.25. Appl., 28.4.24).—Lubricating oil having a green fluorescence is obtained by mixing lubricating oil from the residue of a cracking process operated under such conditions of temperature and pressure as to produce a polymerised and stabilised viscous oil stock, with lubricating oil obtained from petroleum by processes carried out below the stabilising conditions of temperature and pressure. L. A. COLES.

Treating crude oil or petroleum. Treating petroleum. C. G. HINRICHS (U.S.P. 1,566,008–9, 15.12.25. Appl., [A] 23.11.18, [B] 3.11.23).—(A) Natural petroleum emulsions are mixed with a chlorine additive product of a liquid fatty acid in quantity sufficient to effect separation of the oil from brine and other impurities. (B) Water-soluble thiocyanates are added to aqueous emulsions of hydrocarbon oils. L. A. COLES.

Revivifying fuller's earth [used for refining mineral oils]. F. W. HALL, Assr. to TEXAS Co. (U.S.P. 1,562,550, 24.11.25. Appl., 28.7.23).—Spent absorbent used in refining mineral oils is subjected to the distilling action of a carrier gas and then heated in presence of air. H. MOORE.

Treating [spent] decolorising and clarifying hydrous magnesium silicate. M. L. CHAPPELL, Assr. to STANDARD OIL Co. (U.S.P. 1,562,868, 24.11.25. Appl., 15.10.24).—Spent hydrous magnesium silicate which has been used for treating mineral oils can be revivified by treating it with a mixture of acetone, alcohol, and petroleum containing a small quantity of sulphuric acid. T. S. WHEELER.

Bituminous emulsions. G. S. HAY (E.P. 243,398, 31.5.24 and 28.2.25).—Bitumen is heated to 100—110° and mixed with up to 10% of an emulsifying agent comprising starch or other farinaceous material or dextrin, and after agitating, with a dilute solution of alkali. A portion of the starch may be replaced by a fatty acid, or added to a fatty acid in the form of an alkali gel. B. W. CLARKE.

Gasifying and carbonising coal and like substances. H. MACAUX, Assr. to SOC. LYONNAISE DES EAUX ET DE L'ECLAIRAGE (U.S.P. 1,567,967, 29.12.25. Appl., 3.10.22).—See E.P. 186,927; B., 1923, 1164 A.

Process of making motor fuel. L. DE FLOREZ, Assr. to TEXAS Co. (Re-issue 16,239, 29.12.25, of U.S.P. 1,437,045, 28.11.22. Appl., 10.10.24).—See B., 1923, 86 A.

Materials for refining hydrocarbon oils. R. CROSS (E.P. 227,084, 27.10.24. Conv., 31.12.23).—See U.S.P. 1,515,733; B., 1925, 64.

Apparatus for treating hydrocarbons. F. G. Niece, Assr. to Internat. Holding Co. (U.S.P. 1,566,341 and 1,566,416, 22.12.25. Appl.,1.7.22 and 17.7.22).—See E.P. 185,632 and 230,339 ; B., 1922, 850 A ; 1925, 436.

Classification of furnace ash, gas retort ash, household refuse, etc. G. H. Hadfield (E.P. 244,579, 9.12.24).

Crushing ferruginous substances (E.P. 236,578).—See I.

Treating substances centrifugally (E.P. 243,946).—See I.

Obtaining vanadium from petroleum ash (U.S.P. 1,563,061).—See X.

III.—TAR AND TAR PRODUCTS.

Insulating material from sapropel tar. N. Zelinski and B. Maxorov (Neftjanoe i slancevoe Chozjajstvo, 1925, 8, 945—949 ; Chem. Zentr., 1925, II., 2301).—The best conditions for the production of material suitable for the insulation of metal cables, from sapropel tar of d_{50}^{50} 0·9554, viscosity (Engler) 3·20 at 50°, iodine value 98·9, dropping point 30·8°, and containing 2·5—18% of water, are rapid distillation until 40—60% of the tar has distilled over. The residue is a plastic material having d_{50}^{50} 0·996—1·000, dropping point 43—103°, softening point (Kremer–Sarnow) 38—70° ; it is completely liquid at 120°, adheres to sheet metal, and does not crack on bending ; it contains H about 9·5%, C about 85%, O, and traces of N and S, and is free from mineral acids ; and in a layer 2·3 mm. thick it has a dielectric resistance of 33·6 kilovolts in a test extending over 35 secs. L. A. Coles.

IV.—DYESTUFFS AND INTERMEDIATES.

Synthetic indigo. R. Lepetit (Chim. et Ind., 1925, 14, 852).—A process for the production of synthetic indigo, described by the author in a sealed note deposited with the Soc. Ind. de Mulhouse in 1900, and stated to be used by Du Pont de Nemours and Co., consists in treating aniline with formaldehyde and sodium bisulphite. The following reaction takes place : $Ph{\cdot}NH_2 + NaHSO_3 + CH_2O = [H_2O + NHPh{\cdot}CH_2{\cdot}SO_3Na$, and the product is treated with sodium cyanide to form phenylglycine nitrile, $NHPh{\cdot}CH_2{\cdot}CN$, which is subsequently fused with alkali hydroxide. L. A. Coles.

Perylene and its derivatives. Zinke, Springer, and Schmid.—See A., 1926, 71.

Dimethyl*iso*indigotins. Wahl and Faivret.—See A., 1926, 79.

Volumetric determination of nitrobenzene. Kolthoff.—See A., 1926, 84.

Patents.

Manufacture of sulphide dyes and intermediate products. W. Carpmael. From Farbenfabr. vorm. F. Bayer u. Co. (E.P. 243,557, 12.1.25).—Intermediates having the general formula, $C_6H_4\!\overset{NH}{\frown}\!C_6H_3{\cdot}CO{\cdot}NXY$ (X=hydrogen, alkyl, or aryl, Y=alkyl or aryl), which readily condense with nitrosophenols forming indophenols suitable for the production of sulphur dyestuffs, are obtained by condensing carbazolecarboxylic acids with primary or secondary aliphatic or aromatic amines, in the presence of condensing agents. For example, 10·5 pts. of phosphorus trichloride are added to a mixture of 42 pts. of carbazole-2-carboxylic acid, 22 pts. of *p*-toluidine, and 900 pts. of toluene at 70°. After boiling for several hours, toluene is removed by distillation in a current of steam, and 2-*carbazoloic acid* p-*toluidide*, m.p. 294°, is filtered off and dried. A solution of the product in 3200 pts. of 90% sulphuric acid at 0° is mixed with a solution of 27·5 pts. of *p*-nitrosophenol in 450 pts. of 90% sulphuric acid, and, when condensation is complete, the solution is poured on to ice, and 2-*carbazoloic acid* p-*toluidide indophenol* is separated by filtration. The indophenol is sulphurised, *e.g.*, by boiling with alcohol, sodium sulphide, and sulphur under a reflux condenser for several hours, yielding a dyestuff which dissolves in sodium hyposulphite solution giving a yellow vat which yields on cotton fast, bluish-black shades on subsequent oxidation. The preparation of 2-*carbazoloic acid dimethylamide*, thick needles, m.p. 198° (decomp.), 1-*carbazoloic acid 2-carbazolamide*, and 3-*carbazoloic acid* p-*toluidide*, and of the *indophenols* therefrom is also described. L. A. Coles.

Production of [anthraquinone] dyestuff intermediates. J. Thomas, and Scottish Dyes, Ltd. (E.P. 243,505, 2.7.24).—Nitroanthraquinone derivatives substituted in the β-position are produced by condensing benzoylbenzoic acids substituted in the *para*-position in the presence of strong sulphuric acid or weak oleum, and nitrating the anthraquinone derivatives formed by the condensation, without separating them from the solution. The products are subsequently converted into the corresponding amino-compounds and simultaneously purified by treatment with alkaline reducing agents followed by washing with water. For example, a solution of 11·1 pts. by wt. of *p*-chlorobenzoylbenzoic acid, which is prepared by condensing chlorobenzene with phthalic anhydride in the presence of aluminium chloride, in 55·5 pts. of 6% oleum, is maintained at 140° for 2 hrs., and then cooled below 18°, when 3·5 pts. of sodium nitrate are added during ½ hr. with stirring. After keeping for 16 hrs., the melt is boiled with 600 pts. of water, and filtered after further dilution with water. A quantity of the moist product containing 10 pts. of crude nitro-2-chloroanthraquinone is boiled for 1 hr. with a solution of 25 pts. of crystalline sodium sulphide in 450 pts. of water, and the product is filtered off and washed, yielding 9 pts. of amino-2-chloroanthraquinone, m.p.

176—180°. Nitro- and amino-2-methylanthraquinone are prepared in a similar manner from *p*-toluoylbenzoic acid. L. A. Coles.

Production and isolation of alkali salts of aromatic sulphonic acids. H. W. Hillyer, Assr. to Nat. Aniline and Chemical Co. (U.S.P. 1,564,239, 8.12.25. Appl., 3.1.20).—Sodium salts of aromatic sulphonic acids are obtained by treating a solution of the corresponding calcium salt with sodium chloride, whereby the sodium salt is formed and precipitated. A. Cousen.

Indigoid dyestuff of the anthraquinone series and intermediate products. Soc. of Chem. Ind. in Basle, Assees. of B. Mayer and W. Moser (U.S.P. 1,567,158, 29.12.25. Appl., 18.11.24).—See E.P. 225,554; B., 1925, 953.

Aromatic derivative of cyanuric chloride. Soc. of Chem. Ind. in Basle, Assees. of H. Fritzsche and P. Schädeli (U.S.P. 1,566,742, 22.12.25. Appl., 26.3.25).—See E.P. 240,371; B., 1925, 954.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Oxidation of drying oils: effect of anti-oxidising agents. Taradoire.—See XII.

Patents.

Treating flax, hemp, or like textile materials to obtain their fibres in a silky and supple form. C. Dubois (E.P. 232,935, 25.3.25. Conv., 26.4.24).—Fibres of flax, hemp, or the like, after retting, are treated with a hot alkaline lye at the ordinary pressure, washed with water, then with hot soapy, soft water, and rinsed with water. This process is repeated until all resinous substances have been removed, and the fibres are thus rendered supple and silky. The silky lustre and feel may be further improved by impregnating with a suitable oil or varnish. A. Geake.

Washing or otherwise treating wool, silk, soiled fabrics, or other fibrous materials. E. C. Duhamel, and Comp. Gén. des Ind. Textiles (E.P. 243,360, 8.9.24. Conv., 7.9.23; cf. E.P. 221,521, B., 1925, 985).—In order to reduce the loss of soap in cleaning operations, due to absorption by fats, dirt, etc., the soap solution and the material to be treated are passed in opposite directions through a series of vats. After the first hour the liquor squeezed from the material leaving the first vat is run to waste, and replaced by water from the last vat; the consequent dilution of the soap solution causes a part of the absorbed soap to be liberated. A. Geake.

Preparing viscose solutions. E. S. Humboldt, Assr. to Pacific Lumber Co. (U.S.P. 1,562,885, 24.11.25. Appl., 26.11.23).—Raw viscose is suspended or dissolved in water at 50° and heated until a sample coagulates immediately when treated with saturated sodium hydrogen carbonate solution. The whole is then coagulated by treatment with a solution of sodium chloride and sodium hydrogen carbonate, and the precipitate washed and dissolved in dilute caustic soda for spinning. T. S. Wheeler.

Reducing viscosity of nitrocellulose. N. S. Kocher and V. E. Kimmel, Assrs. to Eastman Kodak Co. (U.S.P. 1,564,689, 8.12.25. Appl., 25.2.25).—Nitrocellulose is treated with a solution of a mineral acid and an alkali chlorate, the action being stopped before denitration of the nitrocellulose reduces its nitrogen content below 9·5%. A. Geake.

Removing pyridine from nitrocellulose. L. W. Eberlin, Assr. to Eastman Kodak Co. (U.S.P. 1,564,765, 8.12.25. Appl., 11.2.25).—Solid colloidised nitrocellulose, containing pyridine, is treated with a chlorinating agent in the presence of water and a substance facilitating penetration. When the pyridine is decomposed the nitrocellulose is removed and treated with an anti-chlor. A. Geake.

Manufacture of artificial silk and the like from cellulose derivatives. H. Dreyfus (U.S.P. 1,566,384, 22.12.25. Appl., 10.3.22).—See E.P. 182,166; B., 1922, 627 a.

Recovering lye from disintegrated celluloses. G. Mosebach (U.S.P. 1,567,668, 29.12.25. Appl., 5.5.25).—See G.P. 398,041; B., 1925, 986.

Chlorination of sulphite-[cellulose waste] liquor. A. Schmidt (U.S.P. 1,567,395, 29.12.25. Appl., 30.3.22).—See E.P. 178,104; B., 1923, 765 a.

Impregnating textile fabrics, ropes and the like. V. Lange (E.P. 230,859, 16.3.25. Conv., 14.3.24.)

Process of making artificial sponges [from cellulose esters]. L. Mostny (E.P. 245,049, 3.11.25. Conv., 25.2.25).

Manufacturing paper. C. Bücking (E.P. 237,598, 16.7.25. Conv., 25.7.24).

Membranes of parchment paper (G.P. 418,210).—See I.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Process for dyeing woollen piece goods evenly. Process for printing woollen piece goods by means of direct dyestuffs without steaming. C. Favre (Sealed Notes [a] 1897, 2.4.09, and [b] 2120, 20.9.11. Bull. Soc. Ind. Mulhouse, 1925, 91, 618 and 619—620. Report by H. Wagner. *Ibid*, 620—621).—[a] Uneven dyeing of woollen piece goods in a winch machine is avoided by retarding the rate of absorption of dyestuff by the following method. The pieces are entered into a cold dye-liquor containg sodium carbonate; the temperature of the dye-liquor is then raised to 30° and sufficient sulphuric acid to reduce the alkalinity to one-half, is added, the pieces at this stage being merely tinted. At 35° the dye-liquor is made slightly acid by addition of a further quantity of

sulphuric acid, whereupon dyeing proceeds rapidly. Subsequently dyeing is continued at boiling temperature, the dyed pieces being afterwards washed and dried. For the dyeing of 9 pieces (each 45 yds. in length) of woollen delaine a dye-bath containing 122 g. of Tartrazine, 125 g. of Lanafuchsine SB, 68 g. of Cyanine B, and 8 kg. of sodium carbonate is used, two successive additions, each of 4 kg. of sulphuric acid of 65° B. (*d* 1·82), being made. [B] Woollen pieces are padded with a thickened dye-liquor containing a direct dyestuff and a small quantity of sodium chloride, then dried, and successively passed through two acid baths, the first containing a cold and the second a hot (80°) 0·5% solution of sulphuric acid, and afterwards washed and dried ; the duration of immersion in each acid bath is ½ min. The resulting shades are particularly even. Suitable padding liquors contain 80 litres of water, 20 litres of dextrin thickening, 1 kg. of salt, and 130 g. of Diamine Fast Blue FFB (C), 8 g. of Benzo Fast Red 8DL (By), and 52 g. of Diamine Yellow CP (C), or 350 g. of Diamine Fast Blue FFB, 80 g. of Brilliant Fast Blue 4G (By), and 8 g. of Diamine Yellow CP. Wagner confirms the advantages claimed for these processes. A. J. HALL.

Crêpe effects on delaines. C. FAVRE (Sealed Notes [A] 2236, 31.3.13, and [B] 2266, 28.7.13. Bull. Soc. Ind. Mulhouse, 1925, **91**, 615—616. Report by A. WOLF. *Ibid.*, 616—617).—[A] In carrying out the crêping process previously described (cf. B., 1921, 467 A) the unbleached delaine should be passed directly into the crêping bath, which must be kept vigorously boiling, the previous process of wetting-out in cold water being unnecessary ; subsequent bleaching should be effected by steeping the woollen pieces for several hours in a bath containing sodium peroxide, since bleaching by means of a bisulphite has a deleterious action on the crêped fabrics. Other acids, particularly sulphuric acid, may be used instead of thiocyanic acid for the production of crêpe effects. For example, pleasing minutely crêped delaines are obtained by treating the woollen pieces for ¼ hr. in a boiling liquor containing 1% of sulphuric acid. Delaine fabric usually shrinks during scouring and bleaching processes 1¼% in length and 6% in width, whereas the corresponding shrinkage produced by crêping by means of thiocyanic acid and sulphuric acid is 5¼% and 11%, and 3½% and 11% respectively. [B] The formation of undesirable creases in delaine fabric during crêping is avoided by the use of a crêping bath containing 1000 litres of water and 25 kg. of sulphuric acid maintained at 87·5° ; the pieces (10 of 80 m. each) are successively entered in open width into this bath through a mouthpiece, liquor from the bath being sprayed into the mouthpiece during the entry of the fabric by means of a pump. The pieces are well agitated for 1 hr. in the crêping bath and are then washed and bleached. Prolongation of the duration of crêping increases the minuteness of the crêpe effects. Wolf reports favourably on the processes, but points out that they are expensive in respect of labour and control. A. J. HALL.

Rendering gelatin [in textile fabrics] insoluble at a moderate temperature by means of formaldehyde in the presence of Turkey-red oil. A. BRYLINSKI (Sealed Note 2054, 9.12.10. Bull. Soc. Ind. Mulhouse, 1925, **91**, 611—612. Report by P. BRANDT. *Ibid.*, 613—614).—A solution containing 10% of gelatin, 3% of 40% formaldehyde, and 2—4% of Turkey-red oil (ammonium sulphoricinoleate) remains liquid for 1½ hrs. in the cold, but for a few minutes only at a temperature of 40—50°, the coagulated product being completely insoluble in boiling water. Fabrics are prepared with insoluble gelatin by successive treatment with a solution of gelatin and a solution containing Turkey-red oil and formaldehyde, and afterwards drying, but more lustrous effects are obtained when the order of treatment is reversed. Brandt reports favourably on the process and finds that the formation of a small quantity of hexamethylenetetramine by interaction of ammonia and formaldehyde has no retarding or deleterious effect on the coagulation of the gelatin Turkey-red oil accelerates the coagulation of gelatin by formaldehyde by reason of its dispersing action, and it may be replaced by similar dispersing agents such as sodium sulphoricinoleate, starch, gum Senegal, Protectol (By), and albumin. A. J. HALL.

Clouding point of soap solutions. BRAUN.—See XII.

PATENTS.

Laundering of textile material after bleaching. H. KOHNSTAMM AND CO. (E.P. 243,877, 30.10.24 ; cf. U.S.P. 1,514,067 ; B., 1925, 38).—A solution of an acid and a chloride or other metal salt, having a reducing action due to the metallic constituent, is a good sour and anti-chlor after bleaching. Stannous and titanous chlorides are specially suitable. Sodium silicofluoride or bisulphate or acetic, sulphuric, or phosphoric acid may be used as acid constituent ; the use of oxalic, citric, hydrochloric, and benzoic acids is disclaimed. A. GEAKE.

Weighting silks. A. PEPPER (U.S.P. 1,565,390, 15.12.25. Appl., 14.8.25).—Silk is treated with a solution of stannic chloride, then washed, treated with a solution of sodium phosphate, washed, treated with dilute acetic acid, washed with cold water, treated with a solution of lead acetate, washed, and afterwards treated with a cold solution of sodium phosphate. A. J. HALL.

Treatment [improving the durability] of [weighted] silk. A. G. BLOXAM. From FÄRBEREI- U. APPRETURGES. (VORM. A. CLAVEL UND F. LINDENMEYER) (E.P. 244,282, 6.1.25).—Weighted silk containing free acid deteriorates during storage more rapidly than silk which is neutral or feebly alkaline. Deterioration is prevented by treating silk, after weighting, with an alkaline substance, such as ammonia, trisodium phosphate, a borate, or an organic base. For example, weighted and dyed silk is treated with a 0·5% aqueous solution of soap and ammonia, or trisodium phosphate, or an alkaline solution containing gum tragacanth and β-naphthylamine. The tensile strength of treated and untreated

silk decreased during storage for 4 months in a tropical climate (but protected from direct sunlight) from 19·6 to 19·1 kg. and 20·7 to 10·4 kg. respectively.
A. J. HALL.

Washable black silk piece goods and process for dyeing them. J. SEYER, Assr. to STANDARD SILK DYEING Co. (U.S.P. 1,565,515, 15.12.25. Appl., 24.2.23).—Black silk piece goods fast to washing in lukewarm soap solutions, and to bleeding in cold water, are obtained by immersing the silk in a boiling liquor containing logwood and fustic extracts, then in a boiling bath containing hæmatin extract and soap, subsequently treating successively with hot solutions containing tannic acid and tartar emetic, and removing loose colour by soaping.
A. J. HALL.

Dyeing silk. J. SEYER, Assr. to STANDARD SILK DYEING Co. (U.S.P. 1,565,516, 15.12.25. Appl., 15.3.23).—Tin-weighted silk is dyed in a solution of disodium phosphate containing a direct dyestuff.
A. J. HALL.

Dyeing cellulose acetate. BRITISH DYESTUFFS CORP., LTD., W. H. PERKIN, and C. HOLLINS (E.P. 244,267, 16.12.24).—Dyestuffs suitable for application, without the use of dispersing agents, to cellulose acetate are prepared by acetylation and nitration, with or without subsequent hydrolysis or reduction, of non-sulphonated amino-, diamino-, or poly-amino-anthraquinones or their substituted derivatives. Acetylation may not be replaced by benzoylation since the products have no affinity for cellulose acetate. A brown dyestuff is prepared by the mono-nitration of 20 kg. of diacetyl-1 : 8-diamino-anthraquinone dissolved in 100 kg. of sulphuric acid for 1 hr. at 5—10° by means of 48 litres of a mixed acid containing 200 g. of nitric acid per litre; the crude dyestuff is precipitated by pouring the product into water, and recrystallised from chloro-benzene. A crimson dyestuff is prepared by reduction of mononitrodiacetyl-1 : 8-diaminoanthraquinone by means of stannous chloride. The dinitro-compounds of 1 : 5- and 1 : 8-diacetyldiaminoanthraquinones have no affinity for cellulose acetate but the products obtained from them by hydrolysis or reduction have a useful affinity. Dinitro-β-acetamidoanthra-quinone is a useful brown dyestuff. 2-Nitro- and 4-nitro-anthraquinonylurethanes, the corresponding nitro-amines obtained by hydrolysis, and the amino-urethanes obtained by reduction also have an affinity for cellulose acetate. A. J. HALL.

Dyeing, printing, or stencilling cellulose acetate. BRITISH CELANESE, LTD., and G. H. ELLIS (E.P. 243,841, 23.9.24).—The fastness to light of colours dyed, printed, or stencilled on cellulose acetate is improved by treating the material with a simple or substituted organic amino-compound. The improvement is specially marked when the initial fastness is poor, and with azo dyes. The most suitable compounds are aniline, substituted anilines, alkylanilines, and alkylphenylenediamines; the efficiency increases with the degree of alkylation. Amines, such as diphenylamine, which darken when exposed to air and light are less suitable. The amines may be applied in aqueous solution, as a soluble salt, or solubilised or dispersed. They are readily absorbed by cellulose acetate and only a relatively small quantity is, therefore, required.
A. GEAKE.

Printing or stencilling of products of or containing acetyl cellulose [cellulose acetate]. BRITISH CELANESE, LTD., G. H. ELLIS, and E. GREENHALGH (E.P. 244,143, 28.7.24).—Printed coloured effects fast to rubbing are obtained on cellulose acetate by means of printing pastes containing specially solubilised dyes (cf. E.P. 219,349; B., 1924, 906) or other dyestuffs and a substance capable of exerting a swelling or solvent action on cellulose acetate. Suitable swelling agents include the salts of sulphonic acids of benzene, naphthalene, and anthracene and substituted derivatives such as phenol, naphthol, aminonaphtholsulphonic acids, and particularly a thiocyanate of ammonium or an alkali metal (for alkaline or neutral printing pastes) and zinc nitrate or chloride (for acidic printing pastes). For example, woven or knitted cellulose acetate silk is printed with bluish-red effects by means of a printing paste containing 40 g. of a colour paste (prepared by heating 4 g. of 2 : 4-dinitro-benzene-1-azodimethylaniline, 12 c.c. of water, and 24 g. of 70% sodium ricinoleate until dispersion of the dyestuff is complete), 5 g. of soda ash, 20 g. of ammonium thiocyanate, 40 g. of water, and 295 g. of a thickening (prepared by boiling for 10 min. a mixture containing 600 g. of dark British gum, 600 g. of a 50% aqueous solution of gum arabic, and 800 g. of water); after printing, the fabric is dried (preferably by means of hot air), steamed for 15 to 20 min. in dry steam, soaped lightly at 60°, washed, and dried. (Reference is directed, in pursuance of Sect. 8 (2), of the Patents and Designs Acts, 1907 and 1919, to E.P. 224,925.) A. J. HALL.

Printing textile fabrics. J. R. GEIGY A.-G. (E.P. 223,888, 22.9.24. Conv., 22.10.23).—Instead of ethylene thiodiglycol (cf. E.P. 147,102 and 150,303; B., 1923, 766 A), ethylene glycol and ethylene chlorohydrin are used as colour solvents in the preparation of printing pastes. For example, printing pastes suitable for application to weighted silk contain 4 g. of Chromazurol S conc., 26 g. of water, 8 g. of ethylene chlorohydrin, 54 g. of starch and gum tragacanth thickening, and 8 g. of chromium acetate of 20° B. (*d* 1·16), or 4 g. of Eriochrome Red B, 12 g. of water, 4 g. of 20% ammonia, 10 g. of ethylene glycol, and 70 g. of 50% aqueous gum thickening. Ethylene glycol and ethylene chlorohydrin have no deleterious action on textile materials and may also be used advantageously in discharge pastes.
A. J. HALL.

Cotton fabric having a linen-like finish resistant to wear and washing. E. R. CLARK, Assr. to M. S. ERLANGER (U.S.P. 1,564,943, 8.12.25. Appl., 1.3.23).—Cotton fabric is treated with a viscose solution of low alkalinity, the excess removed,

and the cellulose precipitated from the viscose with a precipitating bath. A. GEAKE.

Treating cotton textiles to impart a linen-like appearance thereto. R. M. L. G. HÉBERT, Assr. to A. VERGÉ (U.S.P. 1,567,264, 29.12.25. Appl., 7.4.25).—See E.P. 232,451; B., 1925, 497. The soured fabric is chlorinated before treatment with sulphuric acid.

Machine for mercerising skeins of thread. C., G., and F. BONNET (E.P. 244,806, 28.6.24).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphatisation of burnt pyrites by the action of the gases obtained on roasting. A. E. MAKOVECKI and K. N. SCHABALIN (Russian J. Chem. Ind., 1925, 1, [3], 21—23).—The conversion of the copper in burnt pyrites into the sulphate by the action of mixtures of sulphur dioxide and air under various conditions has been investigated. The range of temperature most favourable to the formation of copper sulphate in this way is 600—640°, and the extent to which the action proceeds increases with the proportion of sulphur dioxide in the mixed gases. The dissociation pressures of ferrous and ferric sulphates are greater than that of copper sulphate, but the copper rendered water-soluble contains about 20% of water-soluble iron salts. On the industrial scale, the reaction may be carried out in a furnace of the Herreshoff type. T. H. POPE.

Hydrating lime for bleach [bleaching powder] manufacture. D. F. RICHARDSON, W. E. EMLEY, and J. M. PORTER (Chem. Met. Eng., 1925, **32**, 936—937).—A sample of quicklime containing 92·3% CaO and showing 6·6% loss on ignition was slaked with varying proportions of water and then chlorinated. The bleaching powder made was tested for available chlorine at the time of preparation and after storage for 30 days in a stoppered bottle, and also for apparent density. The best results were obtained when little or no free water was present in the slaked lime, *i.e.*, by using 45—50 pts. of water to 100 pts. of quicklime. With these proportions the percentage of $Ca(OH)_2$ in the slaked lime was a maximum (93·6%). C. IRWIN.

Hydrolysis of aluminium sulphate—an application of the quinhydrone electrode. A. J. PELLING (J. Chem. Met. Soc. S. Afr., 1925, **26**, 88—93).—The p_H values of solutions of the salt were determined at various dilutions, using both the Clark shaking hydrogen electrode, and the quinhydrone electrode. The latter was found to be very suitable, and the values obtained agreed well. New platinum must be used for the electrodes. The dissociation constants calculated indicate that at 15° the first stage of hydrolysis is represented by $Al_2(SO_4)_3+2H_2O=2Al(SO_4)(OH)+H_2SO_4$.
S. I. LEVY.

Determination of coloured gases by means of the photo-electric cell, and its application to nitrous vapours. H. and A. COPAUX (Compt. rend., 1925, **181**, 1058—1060).—A method is described for the determination of coloured gases by measuring the intensity of light transmitted through a column of the mixture by means of a photo-electric cell in circuit with a sensitive galvanometer. In the case of nitrogen peroxide mixed with air, the galvanometer deflection was plotted against the percentage of the former gas as determined by chemical analysis. Under the best conditions, differences of 0·02% were detectable in mixtures containing 0·2—0·7% of nitrogen peroxide. The absorption-coefficient (K) was obtained from the relation $K=\log (J_0/J).1/C$, where J_0/J is the ratio of the intensities of incident and transmitted light as shown by the corresponding galvanometer deflections. As the concentration (C) diminishes, K gradually increases, since the sensitivity of nitrogen peroxide to partial pressure changes causes an increase in the coloured form of the gas (NO_2) according to the equilibrium $N_2O_4 \rightleftharpoons 2NO_2$.
J. GRANT.

Absorption of oxygen by pyrogallol. KOVÁCS-ZORKÓCZY.—See A., 1926, 100.

Analysis of mixtures of metallic iron and iron oxides. INGEBERG.—See X.

PATENTS.

Electrode for use in the contact process of making sulphuric acid. F. VORLÄNDER and H. WEBER, Assrs. to A.-G. FÜR ANILIN-FABR. (U.S.P. 1,565,691, 15.12.25. Appl., 29.6.25).—The electrodes of an electrical precipitator for use in the manufacture of sulphuric acid are composed of frame elements carrying a coat of lead, the elements consisting of material which does not react with sulphuric acid with evolution of hydrogen. H. ROYAL-DAWSON.

Utilisation of the gases evolved in the concentration of sulphuric acid for the preparation of sulphuric acid. RHENANIA VEREIN CHEM. FABR. A.-G. (F.P. 592,561, 2.2.25. Conv., 22.2.24).—The gases evolved during the final concentration of sulphuric acid are returned to the lead chamber to be re-converted into acid. A. R. POWELL.

Manufacture of hydrocyanic acid or cyanides [from calcium cyanamide]. STICKSTOFFWERKE G.m.b.H., Assees. of H. FRANCK and H. HEIMANN (G.P. 417,018, 9.4.24).—Calcium cyanamide is treated at a low red-heat with a mixture of hydrogen and carbon monoxide (water-gas), and the hydrogen cyanide evolved is collected and converted into cyanides in the usual way. Alternatively the water-gas may be generated *in situ* by heating a mixture of calcium cyanamide and carbon in an atmosphere of steam. A. R. POWELL.

Oxidation of nitrogen compounds [nitric oxide]. VEREIN FÜR CHEM. U. MET. PRODUKTION (G.P. 418,322, 16.9.22).—Gases containing nitric oxide are brought into contact with highly activated charcoal and removed when the ratio of nitric oxide to nitrogen peroxide is about 1 : 1. A. R. POWELL.

Producing soluble carbonates and hydrates [hydroxides]. A. F. MEYERHOFER (E.P. 243,990, 24.7.24. Addn. to 219,971; B., 1926, 12).—The calcium fluoride produced by interaction of sodium fluoride and calcium carbonate is converted into sodium titanofluoride or other similar double fluoride, instead of into sodium silicofluoride.
C. IRWIN.

Method of hydrating lime. J. P. RICH (U.S.P. 1,565,107, 8.12.25. Appl., 29.12.24).—To a known weight of unground lime the calculated quantity of water is added with agitation. A jet of air is then blown in while mechanical agitation is continued, and the hydrated lime withdrawn by suction. C. IRWIN.

Production of alkali polysulphides containing sulphur in a colloidal form. R. RUSSELL (E.P. 243,394, 23.5.24).—Sulphur, or material containing sulphur, is ground with a compound of sodium or potassium and with a compound of boron together with water. The resulting sludge is heated by steam to 130—140° and filtered. The proportions used may be 2 pts. of flowers of sulphur, 1 pt. of borax, 1 pt. of soda-ash, and 4 pts. of water. A polysulphide solution is thus obtained which contains up to 50% of sulphur by weight and is only slightly alkaline. It is suitable for the cold vulcanisation of rubber, and as a vehicle for sulphur for medicinal purposes. The process is also applicable to the extraction of sulphur from ores, the sulphur being precipitated from the solution by addition of acid. C. IRWIN.

Coating particles [of copper sulphate] with inert powder. L. A. BUNDS, Assr. to SAN JOSE SPRAY MFG. CO. (U.S.P. 1,562,821, 24.11.25. Appl., 14.4.25).—Copper sulphate crystals are ground and mixed with talc powder and the mixture is passed in a current of hot air into a centrifugal separator. The crystals lose water on the surface and become coated with the powder and these heavier particles are separated from the unused talc in the separator. The material thus obtained can be stored without agglomerating and the copper sulphate dissolves readily in water, if required, for example, as an insecticide. T. S. WHEELER.

Apparatus for making anhydrous metal chlorides. F. T. WOHLERS, Assr. to ANHYDROUS METALLIC CHLORIDES CORP. (U.S.P. 1,564,302, 8.12.25. Appl., 17.12.21).—A compound of the metal of which the chloride is required is fed into a rotary retort of fused silica along with a regulated supply of chlorine. The metal chloride is distilled off and condensed. C. IRWIN.

Gas-purifying composition [for removing ammonia from gases]. R. P. MASE, Assr. to MINE SAFETY APPLIANCES CO. (U.S.P. 1,564,433, 8.12.25. Appl., 2.6.22).—Ammonia is removed from gases by means of activated charcoal impregnated with an anhydrous copper salt.

Reducing sulphates and the like. A. H. WHITE, Assr. to J. E. ALEXANDER and E. G. GOODELL (U.S.P. 1,565,300, 15.12.25. Appl., 18.10.24).—Sulphates or other reducible salts are reduced to sulphites by heating in the presence of carbonaceous material and a metal oxide capable of reacting with carbon dioxide at the reduction temperature to form a carbonate, with evolution of heat. The quantity of oxide used is sufficient to form a carbonate with the main part of the carbon dioxide evolved in the reduction, which is effected at a temperature above 300°, but below the temperature at which the carbonate formed is completely dissociated.
H. ROYAL-DAWSON.

Manufacture of sodium sulphide. B. ROOS U. CO. (G.P. 418,313, 16.9.24. Addn. to 404,410; B., 1925, 132).—Sodium sulphate is melted in a shaft or revolving furnace and the requisite coal or reducing gas is then introduced into the fused mass.
A. R. POWELL.

Electrolytic production of copper carbonate. H. HOWARD, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,565,367, 15.12.25. Appl., 29.8.24).—A solution containing an agent supplying carbon dioxide and an electrolyte is electrolysed with a copper anode, enclosed in a diaphragm; carbon dioxide is supplied to the solution during electrolysis, and the copper carbonate produced is removed from the solution by settling or filtering. B. M. VENABLES.

Synthetically made precious stone. D. BERTOLINI, Assr. to SWISS JEWEL CO. (U.S.P. 1,565,777, 15.12.25. Appl., 20.1.25).—A stone having the hardness of natural sapphire and the colour and appearance of natural aquamarine is composed of alumina, and oxides of magnesium, cobalt, and vanadium in the approximate proportion of 86% of alumina, 14% of magnesium oxide, and traces of cobalt and vanadium oxides. H. ROYAL-DAWSON.

Manufacturing hydrogen sulphide. E. BINDSCHEDLER and E. W. RUGELEY, Assrs. to TUBIZE ARTIFICIAL SILK CO. OF AMERICA (U.S.P. 1,565,894, 15.12.25. Appl., 15.4.24).—A mixture of hydrocarbon oils and sulphur is heated and the hydrogen sulphide evolved is purified by passing it through a material of high absorptive capacity.
H. ROYAL-DAWSON.

Apparatus for producing zinc oxide. M. P. KIRK, Assr. to KIRK SIMON SMELTING CO. (U.S.P. 1,566,103, 15.12.25. Appl., 28.3.21).—A zinc compound is mixed with carbonaceous material and fed into the upper end of an inclined rotary kiln, through which hot reducing gases travel in the same direction. The solid residue is discharged from the lower end of the kiln, and the evolved zinc fumes pass into a combustion chamber into which air is passed to form zinc oxide. H. ROYAL-DAWSON.

Manufacturing gypsum from anhydrite. M. FARNSWORTH (U.S.P. 1,566,186, 15.12.25. Appl., 11.3.25).—Anhydrite is ground to a size between the limit in which the largest particles do not substantially exceed 135 microns, with an average size of 18 microns, and that in which the largest particles do not exceed

30 microns, with an average size of 7 microns, and the ground anhydrite is hydrated to form gypsum.
B. W. Clarke.

Manufacture of chromium compounds free from iron. Chem. Fabr. Kunheim u. Co., Assees. of K. Puls (G.P. 418,050, 9.1.24).—Chromic hydroxide suitable for the production of chromium compounds free from iron, is precipatated by the addition of sludge containing ferrous hydroxide or ferrous carbonate to solutions obtained by treating ferrochromium with acids. L. A. Coles.

Manufacture of hydrogen peroxide. Rhenania Verein Chem. Fabr. A.-G., Zweigniederlassung Mannheim, and F. Martin (G.P. 418,321, 16.5.22. Addn. to 403,253; B., 1925, 71).—The crude solution of hydrogen peroxide obtained as described in the chief patent is purified by treatment with hydrofluoric acid or with a soluble phosphate or fluoride.
A. R. Powell.

Manufacture of sodium sulphate and ammonium chloride. E. A. E. Waché (F.P. 589,191, 21.1.24).—The equivalent weight of sodium chloride is added in small portions, with constant stirring, to a solution, at 90—95°, of ammonium sulphate in 1—1·5 times its weight of water. After removing the sodium sulphate which crystallises out, the solution is cooled to 30°. Ammonium chloride separates and is removed. The mother liquor is used for dissolving fresh quantities of ammonium sulphate and sodium chloride. L. A. Coles.

Method of producing chlorine. E. M. Partridge and E. W. Scarritt (U.S.P. 1,566,040, 15.12.25. Appl., 11.5.25).—A pyrosulphate, a chloride, and a permanganate are mixed together in the presence of moisture. H. Royal-Dawson.

Making aluminium sulphate from aluminous materials. R. Moldenke and W. Schumacher (U.S.P. 1,567,610, 29.12.25. Appl., 10.1.23).—See G.P. 383,435; B., 1924, 294.

Evaporating solutions (E.P. 243,982).—See I.

Absorption of gases in liquids (U.S.P. 1,565,366).—See I.

Electrolysis of alkali chlorides (Austr.P. 100,562–3).—See XI.

Ammonium chloride and sulphate (G.P. 417,214).—See XVI.

VIII.—GLASS; CERAMICS.

Machine for transverse tests of clay and glass laboratory specimens. A. C. Harrison (J. Amer. Ceram. Soc., 1925, **8**, 774—783).—A machine capable of giving accurate results for specimens varying widely in size and strength consists essentially of a beam and a lever, which constitute the active element, an adjustable counterpoise, valve mechanism for controlling the flow of shot, and straining mechanism. Results of tests made with the machine on vitreous china and glass are tabulated. F. Salt.

Drying properties of clays. D. C. Lindsay and W. H. Wadleigh (J. Amer. Ceram. Soc., 1925, **8**, 677—693).—Other conditions being constant, the rate of evaporation of water from clay varies approximately in direct proportion to the surface exposed per unit weight of clay. It increases with a rise in temperature, decreases with a rise in the wet bulb temperature of the air, and decreases, after shrinkage water has been removed, with a decrease in the moisture content, due to decrease in the effective vapour pressure of moisture in the material. The most important factor in the drying of clay is the rate of evaporation from the surface in relation to the rate of diffusion of moisture from within to the surface. The causes of the more common drying injuries are explained. Important factors relating to the preparation of drying schedules are the determination of the rate of shrinkage and the point at which shrinkage ceases. A critical stage occurs at, and immediately preceding, the point where shrinkage ceases. Beyond this point, drying may safely be accelerated. Determinations of modulus of rupture on dried specimens of different clays and body mixtures indicated that the strength varied directly with the residual moisture content, the removal of the final 1% of moisture having a marked effect. In an Appendix, a description is given of the special drying cabinet and of the optical flexure balance used. The former is fitted with means for controlling humidity, temperature, and air velocity. Frequent observations of loss of moisture in the specimen are rendered possible by means of a simple weighing device having an extension link into the cabinet.
F. Salt.

New type of oxy-acetylene fusion furnace, with notes on the behaviour of refractories at cone 40. A. F. Gorton and W. H. Groves (J. Amer. Ceram. Soc., 1925, **8**, 768—773).—A small refractory cylinder, made of electrically fused magnesia, is supported on a stand of angle iron. The support, which holds the cone plaque and specimen, rests on square pins, which fit loosely into holes cut through the furnace lining and steel shell 120° apart. New features of the furnace are a slot cut through the front wall to afford a clear view of the cones, and the acetylene burner, which is applied vertically at the bottom of the furnace. The furnace is convenient and economical to operate, and is portable. In the course of a series of tests, it was observed that fireclay and alumina interact at cone 32, alumina and chrome above cone 36, and zircon and magnesia at about cones 37—38. F. Salt.

Articles of fused magnesite [magnesia]. J. Bronn (Chem.-Ztg., 1925, **49**, 1053—1054).—The technical difficulties encountered in the manufacture of articles of fused magnesia are of two kinds; the first is due to the high melting point of magnesia (about 2800°), and the second to the non-plastic nature of the fused material. The addition of other substances (silica, alumina, lime, etc.), with the object of increasing the plasticity, is not practicable, owing to the formation in such mixtures of eutectics of

considerably lower melting point. In the fusion process, magnesite undergoes a certain degree of purification, the secondary ingredients being partially volatilised; for example from a raw material containing 88% of magnesia, the fused material contained 94—95·5%. If the fused product is cooled slowly, well-developed crystals several mm. in length (probably periclase) are formed. The sp. gr. of the fused material is 3·5—3·62 ; that of crucibles made therefrom, 3·56. Such crucibles are now being made in various sizes. They are much more sensitive to abrupt changes of temperature than quartz crucibles, and they also tend to contract slightly when heated. When in direct contact with magnesite or magnesia, carbon has a reducing and volatilising effect at about 2000°. For work at this and higher temperatures, therefore, it is necessary to place the magnesia crucible in a thin carbon or graphite crucible and to fill the space between the two with powdered magnesia. Magnesia crucibles also show great affinity for sulphur, traces of which are present in granulated carbon. F. SALT.

Effect of heat on the strength of calcined kieselguhr–Portland cement mixtures. S. J. MCDOWELL and H. M. KRANER (J. Amer. Ceram. Soc., 1925, 8, 784—788).—The mixture contained 55% by weight of Californian kieselguhr (calcined at 1200°) and 45% of Portland cement. To this dry mixture 65% by weight of water was added. Specimens were moulded from the damp mixture, allowed to age for 28 days, and heated at the rate of 100° per hr. The moduli of rupture were determined on specimens heated at intervals of 100° up to 1100°. A great decrease in strength was noted between room temperature and 100°, and a further decrease between 100° and 200°. Between 200° and 500° the strength was practically constant, but fell again sharply between 500° and 600°. Between 600° and 900° it was fairly constant, a slight increase occurring at the latter temperature. A rapid decrease again took place above 1000°. F. SALT.

Comparison of methods used in estimating the maturing of terra cotta. L. ANDERSON (J. Amer. Ceram. Soc., 1925, 8, 762—767).—A comparative study was made of practical methods, with the object of developing more satisfactory means of determining the maturing point of terra cotta fired in a down-draught kiln. Colour trials (red body dipped in red slip), which were formerly used to indicate temperature by a change in colour, were found unsatisfactory, nor were shrinkage trials entirely satisfactory ; shrinkage was found to continue during the "soaking" period after firing had ceased, and also on re-fired trials. The actual maturing temperature must be determined by the additional aid of cones. Shrinkage trials may, with advantage, be placed in different parts of the kiln, in order to provide useful data regarding variations of temperature within the kiln. F. SALT.

Effects of composition on the properties of sheet steel enamels. H. G. WOLFRAM and W. N. HARRISON (J. Amer. Ceram. Soc., 1925, 8, 735—755).—Three series of enamels were studied, in which the refractory portion was supplied by felspar alone, by a 7 : 5 mixture of felspar and flint, and by flint alone. The frits consisted of 60% of this refractory portion, 5% each of boric oxide and sodium oxide, and 30% of equal proportions of three of the following common constituents: the oxides of boron, sodium, and zinc, cryolite, fluorspar, and sodium antimonate ; these were substituted systematically one for the other. The enamels were applied to 8-inch steel plates, and were tested for resistance to thermal shock, acid attack, and impact, and for relative opacity. The effect of substituting one constituent for another varied to some extent with the type of enamel ; hence definite conclusions are impossible. Enamels most suitable for general use were found among those containing both felspar and flint ; others having special properties occurred in the two series containing felspar only and flint only. Enamels of the former type (felspar only) were more resistant to impact-on-edge, less resistant to impact-on-centre, to quenching, and to acid attack, and less opaque. F. SALT.

Formation of mullite from cyanite, andalusite, and sillimanite. J. W. GREIG (Amer. J. Sci., 1926, [v], **11**, 1—26).—See B., 1925, 761.

PATENTS.

Melting and fining glass. A. L. HARRINGTON, Assr. to PITTSBURGH PLATE GLASS CO. (U.S.P 1,564,235, 8.12.25. Appl., 5.12.23).—A preliminary fusion of the batch in a pot is followed by a transference of the pot to an electric furnace in which melting and fining are completed. A. COUSEN.

Making sheet glass. H. K. HITCHCOCK (U.S.P. 1,564,240, 8.12.25. Appl., 5.2.25).—Against an outlet slot from a tank furnace is a covered bath of molten metal, wider that the slot, and provided with means of regulated heating between cover and bath. Glass flowing from the slot passes over the metal and through a pair of rolls, at least one of which is driven, placed at the forward end of the bath. A. COUSEN.

Preparing quartz rods or canes. L. B. MILLER, Assr. to BRITISH THOMSON-HOUSTON CO., LTD. (E.P. 237,264, 15.7.25. Conv., 18.7.24).—Quartz in the plastic state hanging from a furnace in the form of a rope subject to gravity, is continuously cut into sections by mechanical means and allowed to cool while still hanging freely. B. W. CLARKE.

[Fused silica] refractory material. W. A. HARTY (U.S.P. 1,563,853, 1.12.25. Appl., 18.11.21).—Fused silica is produced by fusing sand in an electric furnace beneath an isolating layer of amorphous or crystalline silicon carbide. F. SALT.

Treatment of clay. T. W. PARKER (E.P. 243,929, 5.2.25).—Clay is purified by deflocculating it in aqueous suspension by means of sodium silicate, together with a non-soap-forming acid (*e.g.*, sulphuric or hydrochloric acid) in amount insufficient to neutralise the alkali content of the silicate, and, after separation, is flocculated by addition of further acid. A. COUSEN.

Preparation of clay. CHEM. LAB. FÜR TONIND. U. TONIND.-ZTG., H. SEGER U. E. CRAMER, and M. PLATZSCH (G.P. 417,361, 13.7.24).—Raw clay is stirred with a solution of the products obtained by treating organic substances, such as wood meal, peat meal, fruit waste, etc., with sulphur dioxide or with waste gases containing sulphur dioxide. The process is applicable to the treatment of non-plastic clays, and the preparation of the clay can be completed within a few weeks. A. R. POWELL.

Melting furnace of the tilting type [for fusing basalt]. P. DHÉ (U.S.P. 1,558,250, 20.10.25. Appl., 1.3.24).—A tilting furnace for the fusion of basalt, providing for the direct casting of the molten material in moulds, is built of firebricks, and is provided with a tap-hole at the front and a loading aperture at the rear. The hearth is in three sections of approximately equal size; that at the rear is inclined toward the front, the other two being inclined toward the rear. A feeding hopper, which can be closed by means of a flap, is attached to the rear of the furnace. F. SALT.

Manufacture of a refractory plastic mass [for crucibles]. MASCHINENFABR. OERLIKON (G.P. 416,766, 24.6.24. Conv., 12.5.24).—The usual mixture of fireclay and graphite is moistened with a solution containing boric acid and borax in the molecular ratio of 4:1. Articles made of this mixture dry rapidly in the air without the formation of fissures, even when drying is accelerated by heat. A. R. POWELL.

Decorating or finishing earthenware or [other] pottery. LOVATT AND LOVATT, LTD., and A. E. LOVATT (E.P. 242,898, 27.5.25).—Colouring matter, such as the oxide of a metal, preferably in powder form, is mixed with a matt medium, also in powder form, composed of about 2 pts. of quartz, 1 pt. of lime, and 1 pt. of clay. The mixture is worked to a viscous mass with liquid gum or other substance. The proportions of matt medium and colouring agent vary according to the strength of the latter. The design is transferred, or printed, with this mixture, upon ware which has been dipped in glaze and dried, but not fired. A single firing operation then produces an unglazed design upon a glazed ground. F. SALT.

Opacifying enamels and glazes. H. KRETZER (G.P. 416,937, 16.5.15).—The vitreous constituent of enamels and glazes is ground with insoluble fluorides of metals which form white oxides and with silica or acid silicates of metals the oxides of which are white. Titania or acid titanates may also be added. Subsequent strong ignition of the mixture improves the covering power. A. R. POWELL.

Refractory material. S. J. LUBOWSKY, Assr. to METAL AND THERMIT CORP. (U.S.P. 1,567,445, 29.12.25. Appl., 18.12.22).—See E.P. 232,679; B., 1925, 548.

Porcelain crucible. A. KÖNIG, Assr. to STAATL. PORZELLAN-MANUFACTUR (U.S.P. 1,567,654, 29,12.25. Appl., 5.8.24).—See E.P. 220,618; B., 1925, 244.

Sintering and melting refractory oxides etc. (G.P. 417,375).—See X.

IX.—BUILDING MATERIALS.

PATENTS.

Treating timber for preventing subsequent shrinkage. SPIRITUS-PRESSHEFE U. CHEM. FABR. HAMBURGER KUFFNER A.-G., and E. MURMANN (E.P. 230,044, 16.2.25. Conv., 27.2.24).—Timber is subjected in closed vessels to the simultaneous action of ammonia (up to 0·5% by weight of the timber) and steam at a temperature not exceeding 120°, preferably 70—100°. B. W. CLARKE.

Protection of wood and other materials against termites and other gnawing insects. TERMIT, LTD., A./S. (E.P. 243,595, 26.3.25. Conv., 17.2.25).—Wood is impregnated or coated with a dilute aqueous solution containing an alum and a small proportion of lead acetate, which interact to form aluminium acetate. The salts are mixed in the dry state, with or without the addition of camphor, volatile oils, or colouring matter. B. W. CLARKE.

Manufacture of cement. T. RIGBY (E.P. 243,410, 28.7, 13.8, 22.10, 3.12, and 5.12.24).—Cement slurry is dispersed in the kiln by spraying devices which are situated outside the kiln head and project the slurry to different regions spaced lengthwise along the kiln. Partly dried material or dry dust carried out in suspension by the kiln gases is collected by a portion of the slurry fed to the kiln as a liquid stream, or by the slurry before dispersion, concentrating this to a water content of 36—37%. The supply of slurry is arranged so that a wetter region is ensured nearer the kiln mouth. B. W. CLARKE.

Alumino-calcareous hydraulic cement. U. B. VOISIN (E.P. 243,876, 30.10.24).—A mixture of bauxite and calcareous material (lime) is heated for 9—12 hrs. at a temperature (900—1100°) well below the melting point in order to avoid partial melting. This prevents adhesion to the walls of the kiln, and produces an alumino-calcareous cement with rapid hardening properties and good resistance to sea water and selenitic waters. B. W. CLARKE.

Paving and surfacing material. C. E. RAMSDEN (E.P. 243,418, 1.8.24 and 7.2.25).—Granular materials possessing a low degree of porosity, *e.g.*, ground flint, slag, or other silicious material, are superficially impregnated with a green colouring matter (chromium compounds) fixed by firing. The resulting material can be rammed or rolled, with or without a binding material consisting of blown linseed oil or rubber latex, to form a surfacing material for hard tennis courts or the like. B. W. CLARKE.

Manufacture of building materials. C. D. BURNEY (E.P. 244,142, 12.7 and 14.8.24.)—Fibrous material, *e.g.*, sawdust, impregnated with a solution of a metal salt, and treated to produce an insoluble

precipitate, is mixed with cement, mortar, etc., and moulded under pressure to form bricks and the like. B. W. CLARKE.

Induration or mineralisation of organic matter. C. D. BURNEY (E.P. 244,178, 12.9.24).—Fibrous organic matter, *e.g.*, sawdust, wood pulp, etc., is impregnated with a solution of a metal salt (excluding highly basic solutions) and treated to produce an insoluble precipitate. Thus, sawdust is treated with 10% ferrous chloride solution and mixed with calcium hydroxide or calcium carbonate; or treated with sodium aluminate and exposed to carbon dioxide; or treated with a mixture of ferric chloride and a soluble dichromate. The mineralised sawdust is mixed with cement, and, on setting forms artificial wood or artificial stone, according to the proportions of sawdust and cement. B. W. CLARKE.

Glazing or surfacing material for tiles, slabs, and other articles. W. J. S. DUFTON and T., W. J., and L. S. OBANK (E.P. 244,312, 27.3.25).—A mixture of water colours, lime and/or cement, and polymerised oils, treated with slightly alkaline water, with or without the addition of infusorial earth, is applied to concrete, tiles, etc., as a cold glazing or facing material. B. W. CLARKE.

Sulphur-containing article. R. F. BACON, W. H. KOBBÉ, and P. H. BASCOM, Assrs. to TEXAS GULF SULPHUR Co. (U.S.P. 1,561,767, 17.11.25. Appl., 25.11.22).—Concrete or burnt clay is immersed in molten sulphur at 125° until it has absorbed about 15% of its weight. The product has a tensile strength at least ten times that of the original concrete or clay. T. S. WHEELER.

Manufacture of artificial stone. J. JAKOB (G.P. 417,360, 23.12.24).—Artificial stone may be made by melting with sulphates silicates of the type R''_2SiO_4, mixtures containing more bases than an equimolecular mixture of R''_2SiO_4 and $R''_2Si_2O_6$, silicates of tervalent metals of the type $R'''_4(SiO_4)_3$, mixtures more basic than an equimolecular mixture of $R'''_4(SiO_4)_3$ and $R'''_4(Si_2O_6)_3$, or mixtures of any of the foregoing classes of compounds. A. R. POWELL.

Cement compositions for making artificial stone and treatment of castings made therefrom. W. J. S. DUFTON and T., W. J., and L. S. OBANK (E.P. 244,973, 27.3.25).

Bituminous emulsions (E.P. 243,398).—See II.

Gypsum from anhydrite (U.S.P. 1,566,186).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of melting scrap on the working of the blast furnace and the economy of cast-iron production. E. BORMANN (Stahl u. Eisen, 1925, **45**, 2041—2049, 2085—2091).—The effect of the addition of up to 81% of scrap to an ore burden was investigated. The coke and lime consumption fell to a minimum at 70% of scrap. The daily output rose to a maximum at 75% of scrap and the amount of slag produced was a minimum at 78%. In practice a saving in coke of about 30% and an increase in production of about 60% was obtained. Heat balances were drawn up for one ore charge and four charges with increasing additions of scrap. As the amount of scrap used rose the carbon monoxide in the waste gases increased and the carbon dioxide fell. The calorific value of the waste gases rose but the degree of efficiency of the fuel used fell. The total heat input and output first increased, then fell. The heat introduced by the hot blast increased, the heat in the waste gases rose, and greater quantities of cooling water were required. The heat losses were larger when scrap was added, but a working balance drawn up for the complete furnace installation showed that the heat consumption in a blast furnace working with scrap was smaller. The temperature and composition of the gases in a furnace working with scrap charges were taken at nine different points starting from the throat to just above the tuyère level. The volume of the gases increased in the tuyère zone and then remained approximately constant to within 4 m. of the throat, when it increased again. Nitrogen was approximately constant throughout, but decreased at the throat. The carbon monoxide, after remaining approximately constant at 60% (per 100 of nitrogen) to within 5 m. from the throat, first increased and then decreased. Carbon dioxide was absent from the tuyères up to 5 m. from the throat, when it increased fairly rapidly. Comparative figures are given for a furnace working with ore. In a blast furnace melting scrap the zone of direct reduction was large and that of indirect reduction small. Curves were obtained showing the total oxygen and carbon, also the excess oxygen and carbon, in furnaces working with ore and scrap. The temperature in the latter rises rapidly from the throat downwards and is considerably higher than in ore furnaces. Reaction equilibria in furnaces working with scrap are discussed and a diagram of reaction zones is given. Variations in the temperature and gas composition in the top zones of scrap-working furnaces during 90-min. pauses were obtained. At the throat the temperature continuously rises, and after a time the carbon monoxide increases and the carbon dioxide falls. T. H. BURNHAM.

Analysis of materials containing a mixture of metallic iron and iron oxides. H. C. M. INGEBERG (Ind. Eng. Chem., 1925, **17**, 1261—1262).—The total iron (metallic iron plus bivalent iron plus tervalent iron) is determined by one of the usual methods. To determine tervalent iron 0·5 g. of the sample is treated with 35 c.c. of $KCuCl_3$-citric acid solution (3 g. of citric acid dissolved in 100 c.c. of a solution containing 560 g. of $CuCl_2,2H_2O$ and 245 g. of KCl per litre), stirring and keeping the temperature below 16°. After keeping for 3 hrs. the residue is washed with cold citric acid solution and three times with hot water and dissolved in hydrochloric acid, tervalent iron being then determined by one of the usual methods. A third portion of the sample

is used to determine the quantity of chlorine (g.-equiv., c, per mg. of sample m) necessary to oxidise metallic iron and bivalent iron present to tervalent iron. 0·3—0·4 g. of the sample is weighed into a Bunsen–Fresenius absorption apparatus and 100 c.c. of $KCuCl_3$–$FeCl_3$ solution (10 g. of $Fe_2Cl_6,12H_2O$ dissolved in 100 c.c. of a solution of 560 g. of $CuCl_2,2H_2O$ and 245 g. of KCl per litre) are added. After 3 hrs., 20 c.c. of concentrated hydrochloric acid are added, the mixture is boiled, and 1 g. of manganese dioxide added. The chlorine evolved is absorbed in a solution of potassium iodide, and the iodine titrated with 0·1N-sodium thiosulphate. Then $(x\times m)/(55\cdot84\times100/3)+(y\times m)/(55\cdot84\times100)=c$, in which $x=\%$ metallic iron, $y=\%$ bivalent iron, and $z=\%$ tervalent iron. H. MOORE.

Electrolytic production of heavy metals from fused electrolytes. I. Iron. F. SAUERWALD and G. NEUENDORFF (Z. Elektrochem., 1925, **31**, 643—646).—Melts made up of 75% of sand and 25% of ferrous oxide (containing ferric oxide) are fluid at 1260°. When these are electrolysed with iron electrodes in externally heated cells at 1·7—2·0 volts or under self-heating conditions at 20—40 volts, there is cathodic deposition of spongy iron, which has a tendency to grow towards the anode. Current efficiencies in the former case were in the neighbourhood of 28%, in the latter of 8%.
W. A. CASPARI.

Density measurements at high temperatures. VI. Change in volume of pig-iron on melting. F. SAUERWALD and J. WECKER (Z. anorg. Chem., 1925, **149**, 273—282; cf. Sauerwald, Allendorf, and Landschütz, B., 1924, 715).—The apparatus previously used requires modification on account of the fact that fused sodium chloride attacks solid white pig iron. This effect is absent with fused white pig iron, and less pronounced with grey pig iron, so that it would appear to be due to solid cementite. Density determinations show that grey pig iron contracts when it melts, whereas white pig iron expands. The contraction is probably due to the formation of carbide in the fused mass.
R. CUTHILL.

Variation with temperature of the resistance to impact of steel. J. COURNOT and K. SASAGAWA (Compt. rend., 1925, **181**, 1065—1066).—The Charpy pendulum has been adapted to the measurement at various temperatures of the work of impact per unit volume and the percentage elongation of extra hard and soft steels. From these data the mean unit tension has been calculated, but no correspondence is shown with the rupture-load of the ordinary tensile tests. Both determined values show maxima at 200°, and minimum values at 500° and 600° for soft and hard steels respectively. These are followed by second maxima at 625° and 700° respectively, and finally by a sharp fall. More complex results were obtained with nickel-steel. J. GRANT.

Rusting of iron. W. KISTIAKOVSKI (Z. Elektrochem., 1925, **31**, 625—631).—Electrodes of iron may exist in five distinct conditions ranging from "superactive" (E_H=0·5—0·9 volt), attainable only in alkaline electrolytes, to "passive" (E_H= −0·2 to −0·9 volt). In the former condition there is an unbroken coating of hydride, in the latter of oxide, and in neither is it possible for the iron to rust. When the coating of oxide is ruptured, which may occur through mechanical or chemical attack, or by crystallisation of the oxide film, local currents are produced and rusting sets in. The acceleration of rusting by carbon dioxide is attributed to its depolarising action in the local currents. When the electrolyte is neutral, *e.g.*, a solution of sodium sulphate (containing dissolved carbon dioxide and oxygen), the formation of sodium carbonate at the passive parts of the metal surface and of ferrous salt at the active parts can be experimentally demonstrated. W. A. CASPARI.

Electrolytic deposition of metals. I. Theory of the mechanism. P. K. FRÖLICH and G. L. CLARK (Z. Elektrochem., 1925, **31**, 649—655).—On a review of the evidence, the authors consider that, on deposition at the cathode, metals do not assume any intermediate state between discharge of the ions and crystallisation of the deposit. Metallic overvoltage is greatest in the case of metals at which hydrogen shows the lowest overvoltage, and *vice versa.* Whereas, therefore, the discharge of ions is a reversible process, metallic overvoltage is an effect due to co-operation of cathodic hydrogen, *e.g.*, by way of transitory hydrides. W. A. CASPARI.

Electrolytic deposition of metals. II. *X*-Ray investigation of electrolytic nickel. G. L. CLARK and P. K. FRÖLICH (Z. Elektrochem., 1925, **31**, 655—658).—Deposits upon platinum and aluminium from electrolytes of various compositions have been examined by means of slit and pinhole beams of molybdenum $K\alpha$ radiation and by the Laue method with white tungsten radiation. For a fibrous or oriented structure in the deposited nickel, low current density, low temperature, and the presence of gelatin are favourable; evolution of hydrogen is unfavourable. The orientation of the structure is parallel to that of the cathode metal in the case of platinum; upon aluminium, however, the deposit shows random orientation, probably due to a film of aluminium oxide. W. A. CASPARI.

Reduction of ferric oxide and iron ores by hydrogen. H. KAMURA (J. Iron and Steel Inst., 1925, **112**, 279—298).—See B., 1925, 804.

Ancient iron from Richborough and Folkestone. J. N. FRIEND (J. Iron and Steel Inst., 1925, **112**, 225—237).—See B., 1925, 806.

Dendritic segregation in iron–carbon alloys. A. SAUVEUR and V. N. KRIVOBOK (J. Iron and Steel Inst., 1925, **112**, 313—321).—See B., 1925, 806.

Influence of strain and of heat on the hardness of iron and steel. A. SAUVEUR and D. C. LEE (J. Iron and Steel Inst., 1925, **112**, 323—329).—See B., 1925, 810.

Retained austenite. J. A. MATHEWS (J. Iron and Steel Inst., 1925, **112**, 299—312).—See B., 1925, 807.

Equilibrium at high temperatures in the iron–carbon–silicon system. M. L. BECKER (J. Iron and Steel Inst., 1925, **112**, 239—253).—See B., 1925, 806.

Chromium–iron equilibrium in carbides recovered from annealed 2·23% chrome steels. E. D. CAMPBELL and J. F. ROSS (J. Iron and Steel Inst., 1925, **112**, 255—265).—See B., 1925, 807.

Iron–nickel and iron–cobalt equilibrium diagrams. L. GRENET (J. Iron and Steel Inst., 1925, **112**, 267—278).—See B., 1925, 808.

Sulphatisation of burnt pyrites. MAKOVECKI and SCHABALIN.—See VII.

PATENTS.

Manufacture of [steel alloy] articles hardened in their marginal layers by nitrogenisation. G. BREWER. From F. KRUPP A.-G. (E.P. 243,613, 16.5.25).—Articles, the external layers of which are to be hardened by nitrogenisation, are made from steel alloys which contain 0·5—2% Al, also silicon, manganese, nickel, chromium, molybdenum, tungsten, vanadium, titanium, or zirconium, either separately or together, in quantity amounting to 0·5—4%, and up to 0·6% C. C. A. KING.

Decarbonisation of cast iron or other metals. P. J. MARTIN and G. F. BERTELS (E.P. 244,204, 3.10.24).—Cast iron is decarbonised by heating with carbon dioxide under pressure. The carbon monoxide formed is re-oxidised to carbon dioxide by means of a regenerating agent, such as a metal oxide, which is included in the system.

L. M. CLARK.

Process of making malleable iron castings. W. J. MILES, JUN. (U.S.P. 1,564,437, 8.12.25. Appl., 10.1.20).—Pig iron is purified partially by melting in a cupola and the molten iron is caused to impinge in a fine stream out of contact with the atmosphere on to the hearth of an air furnace.

C. A. KING.

Electroplating [iron with nickel]. W. R. KING, Assr. to HANSON AND VAN WINKLE CO. (U.S.P. 1,564,581, 8.12.25. Appl., 3.5.24).—Metals subject to corrosion, *e.g.*, iron, are first coated with a nickel-zinc alloy and then with pure nickel.

A. R. POWELL.

Treatment of iron sulphide ores containing other metals. A. T. K. ESTELLE (U.S.P. 1,565,353, 15.12.25. Appl.,12.6.25).—Iron sulphide ore containing other valuable metals, after suitable preliminary treatment, is leached with dilute sulphuric acid. Copper is removed from the solution and ferrous sulphate obtained by crystallising. The impure crystalline mass is treated with ammonia vapour and air and from the dried ferric hydroxide, ammonium sulphate is washed out and separated from accompanying metals (zinc, nickel, etc.). L. M. CLARK.

Cupola furnaces. A. POUMAY (E.P. 232,630, 18.4.25. Conv., 18.4.24).—A cupola furnace is constructed with three sets of tuyères for the air blast, one for directing air to the melting zone, a second higher one introducing air in a gyratory manner, and a third arranged spirally around the cupola above the last-mentioned set and supplying air to the red-hot fuel. By these means the consumption of fuel is reduced as compared with the usual type of cupola. L. M. CLARK.

Metallurgical furnaces. A. BREITENBACH (E.P. 243,402, 25.6.24).—In a furnace for smelting metals, in particular iron, of the type in which a collecting chamber for the molten metal is connected with a melting chamber, heating gases are caused to impinge in an inclined direction on to a shallow depth of molten metal at one end of the collecting chamber so as to stir the metal thoroughly. Towards the other end of the chamber the molten metal collects in a deeper pool, wherein separation of slag is effected. Between the shallow and deeper regions the collecting chamber is restricted by means of a roof inclined downwards, and the heating gases are directed by the roof on to the surface of the metal, which is large in relation to the depth. The waste gases from the melting hearth may be utilised to heat the collecting chamber, the two chambers being so arranged in relation to one another that the gases flow in the collecting chamber at an angle to the direction of flow of the gas in the melting chamber. C. A. KING.

Manufacture of rods, blocks, and the like consisting of very large crystals of highly refractory metals such as tungsten. N. V. PHILIPS' GLOEILAMPENFABRIEKEN (E.P. 235,213, 5.6.25. Conv., 6.6.24).—When tungsten powder is compressed into rods, the size of the crystals formed depends on the pressure. The curve representing crystal size as a function of the pressure shows a maximum for a definite value of the pressure, the position of the maximum depending on the initial state of the tungsten powder. For example, with tungsten powder in which the size distribution is 10—20μ, 8%; 6—10μ, 35%; 3—6μ, 26%, and less than 3μ, 31% of crystals of more than 1 cm. size are obtained by pressing under a pressure of 884 kg./cm.^{2} L. M. CLARK.

[Aluminium–copper] alloys. BRIT. ALUMINIUM CO., A. G. C. GWYER, and H. W. L. PHILLIPS (E.P. 243,405, 19.7.24).—The structure of aluminium–copper alloys, composed mainly of aluminium, with or without other metals, is varied by the addition of a "modifying" agent to the molten alloy. The "modifying" agent may be an alkali oxide or hydroxide, preferably sodium hydroxide, an alkaline-earth metal, a fluoride, or sodamide. For each agent there is an optimum quantity, which yields the best results. For example, in modifying an alloy composed of 92% Al and 8% Cu by means of calcium the optimum quantity is 0·3% Ca. The best percentage in the case of sodium hydroxide and sodium fluoride is usually about 5%, but is dependent

on the composition of the alloy and the cooling rate (cf. E.P. 210,517 and 219,346; B., 1924, 262, 793).
M. Cook.

Treating molten metals [aluminium-bronze] with calcium-copper alloys. W. G. Harvey, Assr. to Amer. Magnesium Corp. (U.S. 1,563,188, 24.11.25. Appl., 10.9.21).—Molten aluminium-bronze is deoxidised by adding a brittle alloy of calcium and copper having a melting point substantially lower than that of the treated metal.
T. H. Burnham.

Producing calcium-copper alloy. W. G. Harvey, Assr. to Amer. Magnesium Corp. (U.S.P. 1,563,187, 24.11.25. Appl., 13.8.21).—A molten calcium salt is electrolysed in the presence of a solid copper cathode until sufficient calcium has been taken up by the cathode for it to become molten in the presence of the molten salt.
T. H. Burnham.

Process for de-oxidisation, and for the protection from oxidation, of metals [during melting]. O. Junker (E.P. 243,998, 8.6.25).—Metals indifferent to carbon are covered with a layer of lampblack or soot during melting or heating processes. Carbon in this form is very finely divided and does not occlude oxygen, and it forms an effective barrier against penetration of oxygen into contact with the metal. C. A. King.

Forming metallic compositions [alloy filaments]. H. Boving, Assr. to Western Electric Co. (U.S.P. 1,562,202, 17.11.25. Appl., 1.6.22).—Nickel, platinum, or tungsten is formed into a suitable shape, such as a filament, and heated in a closed chamber in an atmosphere of an inert gas. When a suitable temperature is attained the vapour of an alkaline-earth metal is led into the chamber. The temperature must not exceed the melting point of the alloy formed. T S. Wheeler.

Tin-zirconium alloy. H. S. Cooper, Assr. to Kemet Laboratories Co. (U.S.P. 1,562,540, 24.11.25. Appl., 27.6.22).—A pyrophoric alloy containing zirconium and 20—40% of tin is claimed.
H. Moore.

Alloy. C. E. Hansen (U.S.P. 1,562,958, 24.11.25. Appl., 6.4.22).—An alloy contains more than 50% of gold, substantial amounts of nickel and iron, and a smaller amount of manganese or magnesium sufficient to increase the tensile strength and workability of the alloy. T. H. Burnham.

Obtaining vanadium values etc. [from petroleum]. E. L. Baldeschwieler, Assr. to Standard Development Co. (U.S.P. 1,563,061, 24.11.25. Appl., 4.8.23).—Petroleum ash is treated with an alkaline reagent to extract the vanadium.
T. H. Burnham.

Electroplating with metallic cadmium and bath therefor. Cadmium-plating. C. M. Hoff, Assr. to Grasselli Chemical Co. (U.S.P. 1,563,413—4, 8.12.25. Appl., [a] 17.1.24, [b] 6.10.24).—(a) A bath for obtaining electrolytic cadmium deposits comprises a solution containing a cyanide, a cadmium compound, and an ammonia compound. (b) A cadmium-plating bath contains a cadmium compound, a substance which tends to inhibit the co-deposition of zinc, an alkali, a cyanide, and a substance which tends to improve the quality of the deposits.
A. R. Powell.

Coating with metals. A. Z. Pedersen, Assr. to Madsenell Corp. (U.S.P. 1,564,710, 8.12.25. Appl., 14.5.20. Renewed 10.11.23).—The metal surface to be coated is cleaned by making the object the anode in a bath of sulphuric acid of more than 75% strength. M. Cook.

Treatment of argentiferous sulphide ores. F. E. Elmore, Assr. to N. C. Christensen (U.S.P. 1,565,072—3, 8.12.25. Appl., [a] 26.3.21, [b] 9.6.22).—(a) Argentiferous lead-zinc sulphide ore is mixed with sodium chloride and heated in the presence of air above 400° but not high enough to cause volatilisation of lead as halogen compound. The zinc sulphide remains mostly unattacked and the oxidised mixture is extracted with strong sodium chloride solution, after which silver is extracted from the residue. (b) After leaching the heated ore with acid brine, the argentiferous zinc sulphide residue is leached with acid chloride brine solvents which extract the silver and leave zinc sulphide undissolved (cf. E.P. 162,026; B., 1921, 435 a). M. Cook.

Solder. J. B. Speed and A. H. Falk, Assrs. to Western Electric Co. (U.S.P., 1,565,115, 8.12.25. Appl., 11.1.24).—A solder containing 11% Ag and 86—89% Sn is claimed. M. Cook.

Alloy for electrical contacts. L. A. Gardner, Assr. to Amer. Telephone and Telegraph Co. (U.S.P. 1,565,358, 15.12.25. Appl., 19.2.24).—The alloy is composed chiefly of gold, together with nickel, zinc, and copper in successively decreasing proportions. L. M. Clark.

Treating [zinc-]coated materials. E. Cornell, jun., Assr. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,565,420, 15.12.25. Appl., 21.12.23).—Zinc-coated materials prior to being worked are heated to a temperature sufficient to change the grain structure of the zinc.
L. M. Clark.

Process by which aluminium and its alloys may be applied to ferrous metal. C. W. Pfeil (U.S.P. 1,565,495, 15.12.25. Appl., 1.5.22).—Aluminium is very finely divided in a bath of an air-excluding, film-producing material, and the compound thus formed is applied to the metal to be coated and is then heated. L. M. Clark.

Process for making and combining "corrode-resisting" metals. C. W. Pfeil (U.S.P. 1,565,496, 15.12.25. Appl., 20.9.22).—A tin-coated ferrous metal is heated until the tin melts and the molten tin is saturated with finely divided aluminium.
L. M. Clark.

Zinc-condenser. H. A. Grine, Assr. to Grasselli Chemical Co. (U.S.P. 1,565,928, 15.12.25. Appl., 3.4.24).—A composition for zinc condensers comprises broken zinc condenser, zinc ore, and a refractory bonding material. L. M. Clark.

Refining lead. P. W. Davis (U.S.P. 1,566,078, 15.12.25. Appl., 14.5.25).—Lead is refined by exposing molten lead alloys to the action of molten litharge, both being in bodies of small dimensions. L. M. Clark.

Continuous sintering and melting of highly refractory metal oxides, ores, etc. Rombacher Hüttenwerke, and J. I. Bronn (G.P. 417,375, 13.3.23. Addn. to 368,328; B., 1923, 508 a).—The charge is passed through a tunnel furnace having a knee-shaped moving hearth heated by means of an arc which moves in the same direction as the hearth. The furnace is especially suitable for the treatment of very viscous materials which tend to adhere to the walls and hearth. A. R. Powell.

Smelting reacting mixtures of oxides and sulphides in suspension. F. Krupp, Grusonwerk A.-G. (G.P. 417,458, 23.2.24).—For the oxidation of sulphides during smelting, solid oxides, sulphates, nitrates, or silicates are added to the charge and for the reduction of oxides, sulphates, nitrates, or silicates, solid sulphides are added. In this way better utilisation of the heat of reaction is obtained, as the volume of gas evolved is reduced. A. R. Powell.

Bearing metal. L. Kaul and E. Haberer (G.P. 417,742, 9.3.24).—Refined zinc is treated in an electric furnace first in the liquid and then in the gaseous state, with complete exclusion of oxygen and nitrogen, whereby a "zinc-nitrogen-hydrazine" compound is formed. This is melted with zinc and tin to form an alloy containing 67·93% Zn, 10·18% Sn, and 21·89% of "zinc-nitrogen-hydrazine." A. R. Powell.

Desulphurisation and concentration of sulphide iron ores. W. S. Millar (U.S.P. 1,567,378, 29.12.25. Appl., 10.3.25).—See E.P. 236,256; B., 1925, 725.

Extracting or refining magnesium. B. E. F. Rhodin (E.P. 234,074, 27.4.25. Conv., 13.5.24).—See U.S.P. 1,539,955; B., 1925, 598.

Alloy. E. Haagn, Assr. to W. C. Heraeus, G.m.b.H. (U.S.P. 1,566,534, 22.12.25. Appl., 21.8.24).—See E.P. 230,356; B., 1925, 363.

Making sheet copper electrolytically. M. A. Jullien (U.S.P. 1,567,737, 29.12.25. Appl., 4.11.24).—See E.P. 244,916; B., 1925, 288.

Reducing ores and producing cement. A. Ferguson (U.S.P. 1,567,934, 29.12.25. Appl., 13.3.25).—See E.P. 235,606; B., 1925, 677.

Metal-melting furnace [provided with agitator]. L. Hall (E.P. 244,915, 24.12.24).

Precipitating and filtering (E.P. 242,383).—See I.

Electric furnace (U.S.P. 1,562,825 and 1,564,800).—See XI.

XI.—ELECTROTECHNICS.

Transformer oils. Staeger.—See II.

Insulating material from sapropel tar. Zelinski and Maxorov.—See III.

Determination of coloured gases by the photo-electric cell. Copaux and Copaux.—See VII.

Rusting of iron. Kistiakowsky.—See X.

Patents.

Removing coatings from conductors. Brit. Thomson-Houston Co., Ltd., Assees. of H. P. Hollnagel (E.P. 232,257, 14.4.25. Conv., 11.4.24).—Sheaths or coatings of insulating material are removed from electrical conductors by immersion in a bath of fused alkali. If a long length of wire is to be treated it is unwound from a reel and passed through a bath containing fused alkali, which must be carefully freed from water but need not be chemically pure. The temperature should not exceed 700°. To strip the ends of conductors these are dipped for a few seconds in fused alkali at 400—600° and subsequently washed in a spray of water. When it is desired to "tin" the bared ends of the conductors, they are dipped without washing into molten solder or other metal capable of alloying with the conductor, preferably through a thin layer of molten alkali. M. E. Nottage.

Electric furnace. G. Evreynoff (U.S.P. 1,562,825, 24.11.25. Appl., 11.11.24).—A furnace for the treatment of steel or the like consists of a carbon crucible forming one of the electrodes and a carbon rod vertically above it which constitutes the other electrode. The material in the crucible is heated by an arc passing from the rod to the material and through it to the crucible. The arc is given a helical form and caused to rotate by means of a coil through which a current is passed. This stabilises the arc and renders its control more easy. T. S. Wheeler.

Electric furnace. M. Unger, Assr. to Gen. Electric Co. (U.S.P. 1,564,800, 8.12.25. Appl., 12.3.24).—The charge is heated by the passage of the current in a crucible providing a relatively wide channel which has a deep narrow groove at the bottom for increasing the hydrostatic head of molten metal. M. E. Nottage.

Electrolytic cell. M. W. Cowles (U.S.P. 1,564,406, 8.12.25. Appl., 2.5.24).—In a cell comprising a number of units, each of which consists of an anode and cathode separated from each other, a device is provided by means of which the electrolyte may be fed into the space between the anode and cathode of each unit independently of the other units. M. E. Nottage.

Electrolysis of alkali chlorides. M. BAMBERGER and J. NUSSBAUM (Austr. P. [A] 100,562, 12.2.17, and [B] 100,563, 7.8.17).—Alkali chloride electrolysis is effected with the aid of a horizontal filter diaphragm composed of (A) asbestos coated with a gel, *e.g.*, of magnesium hydroxide or of an alkaline-earth silicate, or (B) a partially peptised or partially flocculated gel. D. F. TWISS.

Production of electrical incandescence heating material of carbon. DRAHTLOSE HEIZ- U. WIDERSTANDSKÖRPER FÜR ELEKTRIZITÄT G.M.B.H. (G.P. 416,776, 4.7.24).—A long-lived heating element is formed by coating the inner wall of a fireproof tube with a conducting mixture of graphite and a carbonate of a heavy metal; the tube is kept air-tight by stoppers which also serve for effecting electrical contact. The carbon dioxide which is liberated inside the tube when a current is passed protects the conducting material against oxidation. D. F. TWISS.

Iron hydride resistances for regulating electric currents. SÜDDEUTSCHE TELEFON-APPARATE-, KABEL- U. DRAHTWERKE A.-G. (G.P. 417,810, 10.5.23).—Resistance wire produced from electrolytic iron which has been melted in a vacuum is maintained at a red heat in a hydrogen atmosphere until saturated with the gas; such wire shows constant behaviour in use. D. F. TWISS.

Method of making cores for cathodes of vacuum tubes. H. T. REEVE, Assr. to WESTERN ELECTRIC Co. (U.S.P. 1,565,200, 8.12.25. Appl., 12.10.20).—A core for an electron-emitting cathode is made by pressing a mixture of powdered platinum and nickel in the desired proportions into a coherent ingot, heating the ingot to a sintering temperature in the absence of oxidising conditions, and cold-working the ingot into filamentary form. At intervals the work should be annealed at high temperature, the early stages of the cooling being done in the absence of oxidising conditions. M. E. NOTTAGE.

Electrolytic cell. A. E. KNOWLES (U.S.P. 1,566,543, 22.12.25. Appl., 3.4.24).—See E.P. 219,381; B., 1924, 838.

Incandescence lamp. G. R. FONDA, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,566,848, 22.12.25. Appl., 26.1.22).—See E.P. 201,629; B., 1923, 919 A.

See also pages 113, **Preventing adhesion of scale** (E.P. 243,415). 125, **Electrode for making sulphuric acid** (U.S.P. 1,565,691). 126, **Copper carbonate** (U.S.P. 1,565,367). 128, **Melting and fining glass** (U.S.P. 1,564,235); **Fused silica** (U.S.P. 1,563,853). 133, **Alloy filaments** (U.S.P. 1,562,202); **Alloy for electrical contacts** (U.S.P. 1,565,358). 134, **Sintering and melting oxides etc.** (G.P. 417,375).

XII.—FATS; OILS; WAXES.

Rapid oxidation of drying oils; effect of nti-oxidisIng agents. F. TARADOIRE (Compt. rend., 1926, **182**, 61—63).—Cotton treated with a mixture of a drying oil, turpentine oil, and a heavy-metal resinate ignites spontaneously after exposure to air. Untreated cotton is spontaneously inflammable at 210°, and the temperature of asbestos fibre treated with a mixture of drying oil, turpentine oil, and heavy-metal resinate rises to 300°. The spontaneous ignition of cotton waste similarly treated is retarded by treatment of the material with 1% of phenol, β-naphthol, or quinol, and prevented by treatment with guaiacol, α-naphthol, aniline, dimethylaniline, diphenylamine, quinhydrone, hexamethylenetetramine, or sulphur (cf. A., 1924, i, 635). L. F. HEWITT.

Stabilising agents for oils. E. DE' CONNO, E. GOFFREDI, and C. DRAGONI (Annali Chim. Appl., 1925, **15**, 475—484).—Experiments made with linseed and olive oils show that phenol, resorcinol, quinol, and pyrogallol, far from acting as anti-oxygens, actually accelerate the autoxidation of these oils. This oxidation is retarded, but not inhibited, by salicylic or benzoic acid, whereas complete stabilisation of non-drying and drying oils is effected by acetic and glycollic acids respectively, the action of these acids being quite comparable with the catalytic action of phenols in stabilising acraldehyde T. H. POPE.

Analysis of waxes. New chemical constant. R. ROUSSEAU (Bull. Sci. pharmacol., 1925, 449; Ann. Chim. Analyt., 1925, **7**, 355—357).—The saponification values of the crude wax, of the wax after acetylation, of the unsaponifiable matter, and of the unsaponifiable matter after acetylation, are determined. The difference between the first two values gives the acetyl value of the free alcohols, and the difference between the last two values the acetyl value of the total alcohols; the acetyl value of the esterified alcohols is then obtained by difference. The ratio of the acetyl value of the esterified alcohols to the acetyl value of the total alcohols is of use for differentiating various waxes. It amounts to about 0·84 for beeswaxes and crude wool wax ("suintine"), 0·96 for spermaceti, 0·6 and 0·5 for carnauba and raphia waxes, and 0·8 for candelilla wax. D. G. HEWER.

Determination of the clouding point of soap solutions. K. BRAUN (Chem.-Ztg., 1925, **49**, 1012).—The clouding point is defined as that temperature at which a soap solution containing 3% of fatty acids combined with alkali becomes turbid. In washing textiles, if the clouding point lies above the temperature of the washing water, soap is precipitated on the fibres and interferes with subsequent dyeing or printing. Apparatus for determining the clouding point comprises a jacketed beaker fitted with a thermometer and stirrer, and placed in a vessel filled with hot water. The jacket has a narrow vertical slit in one side and a slit three times as wide with a narrow strip down the middle on the opposite side. Soap solution heated above its clouding point is poured into the beaker and viewed through the narrow slit, illumination being provided

behind the wider slit, and the temperature is read at which cloudiness first begins to appear.

L. A. COLES.

isoOleic acid and other unsaturated fatty acids formed by distillation of ι-hydroxystearic acid. VESELY and MAJTL.—See A., 1926, 47.

Chaulmoogric and hydnocarpic acids. SHRINER and ADAMS.—See A., 1926, 47.

See also pages 117, **Petroleum hydrocarbons from fats** (MARCUSSON and BAUERSCHÄFER). 136, **Colour of oils etc.** (WILBORN). 140, **Coconut oil** (HANUS and KOMOROUSOVÁ; LUKAS; BERTRAM and OTHERS.)

PATENTS.

Removing phosphatides from vegetable oils. H. BOLLMANN (E.P. 243,643, 20.7.25. Conv., 15.5.25).—The oils are treated with barium hydroxide. For example 90 pts. of soya-bean oil are shaken with 10 pts. of a 0·1% barium hydroxide solution and the oil is subsequently separated from the aqueous mixture. The removal in this manner of the phosphatides prevents the formation of troublesome emulsions during purification of the oils.

H. M. LANGTON.

Removing free sulphur from [wool] grease. H. CHRISTISON and C. L. NUTTING, Assrs. to ARLINGTON MILLS (U.S.P. 1,561,911, 17.11.25. Appl., 3.6.21).—Grease from raw wool is boiled with a solution of sodium sulphite, when the sulphur present forms sodium thiosulphate and is removed. The grease is then separated from the solution by centrifuging.

T. S. WHEELER.

Saponaceous cleansing compositions. K. HAAS (E.P. 243,423, 22.8.24).—A mixture of a protein (casein, glue) and fat is saponified with excess of alkali, whereby partial hydrolysis of the protein results. When evolution of ammonia becomes distinctly noticeable, formaldehyde or similar substance is added so as to form *in situ* hexamethylenetetramine, which prevents further hydrolytic dissociation of the protein. A product is obtained having the appearance of good household soap and with enhanced washing and lathering properties due to the soap containing 15% of stable proteins.

H. M. LANGTON.

Making emulsions using emulsifiers derived from wool fat. O. HERZOG (E.P. 244,598, 10.1.25).—See U.S.P. 1,543,384; B., 1925, 680.

Membranes of parchment paper (G.P. 418,210).—See I.

Adding A-vitamins to oils (E.P. 243,907).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Determination of solvent in paints, varnishes, etc. H. WOLFF (Farben-Ztg., 1925, 31, 746—747).—Owing to the retention of relatively high-boiling solvents by the non-volatile portion of a paint or varnish, the usual steam-distillation process for solvent recovery requires the use of unwieldly receivers or careful attention over a lengthy period to avoid overflow. The author uses a receiver in which the distilled solvent collects over water which continually drains away *via* a suitably bent side arm sealed on to the main tube. When no obvious alteration in the amount of solvent collected can be seen, the receiver is changed for a smaller tube of the same type, the narrow bore of which renders the presence of very small quantities of solvent apparent. The solvent is either run off and weighed or else its volume is read *in situ*. The volatile medium is held with abnormal tenacity by some paste paints, particularly those containing basic pigments, and the absence of non-aqueous distillate after ¼ hr. (by which time the bulk of the solvent will normally have distilled) may give a misleading impression. Where such cases are recognised the addition of a moderate quantity of hydrochloric or sulphuric acid will facilitate removal of the solvent by breaking down the colloid system.

S. S. WOOLF.

Determination of the colour of varnishes, oils, etc. F. WILBORN (Farben-Ztg., 1925, 31, 747).—The author claims that the use of aqueous iodine–potassium iodide solutions provides satisfactory matches for all the products in the varnish industry the colour of which is of importance, *i.e.*, the paler oils and varnishes. More concentrated iodine solutions afford less satisfactory comparison with the darker products, but the dichromate–sulphuric acid standards used in America, while giving better colour matches, are prone to colour change due to reduction caused, *e.g.*, by dust particles present inadvertently. Fonrobert's "colour numbers" (B., 1925, 931) are considered unsuitable, since two matched samples may show large divergences on parallel treatment either as to dilution or viewing in different thickness of layer. Any method involving liquid "wedges" in hollow prisms must be rejected on this score and the use of standard glass tubes is insisted upon. The permanence of iodine solutions leaves much to be desired, but there is no objection to the use of more permanent comparison liquids, provided that these are first standardised against iodine.

S. S. WOOLF.

Oxidation of drying oils: effect of anti-oxidising agents. TARADOIRE.—See XII.

PATENTS.

Hard rubber varnishes. W. J. MELLERSH-JACKSON. From MECHANICAL RUBBER CO. (E.P. 243,966,84. 25).—A varnish of vulcanised rubber. is obtained by introducing 15—32% of sulphur (calculated on the rubber) into a solution of rubber in a high-boiling solvent, such as solvent naphtha, and heating, *e.g.*, at 163°, until substantially the whole of the sulphur has gone into combination. New rubber may be used or vulcanised scrap rubber; fillers may be added and also an accelerator of vulcanisation. By concentration it is possible to

obtain a fluid varnish with a solid content up to 80%. Paper, fabrics, and similar materials treated with such varnish are rendered resistant to the action of water and have good mechanical and dielectric characteristics. D. F. TWISS.

Waterproof paints. LATEX DEVELOPMENTS, LTD., and R. RUSSELL (E.P. 244,150, 3.9.24).—A waterproof paint which can be applied with a brush without risk of coagulation and, when dry, is resistant to atmospheric influences, is produced by blending a mixture of casein, borax or soda ash or similar alkali, phenol, and ammonia with the desired wet-ground filler or pigment, *e.g.*, lithopone, and with preserved latex, some or all of the casein mixture being introduced into the latex before the addition of the pigmentary material. D. F. TWISS.

Synthetic resin composition. A. E. ALEXANDER. From J. S. STOKES (E.P. 243,470, 9.9.24).—Synthetic resins of superior quality to known phenol–formaldehyde condensation products are obtained by condensing furfuraldehyde with phenolic substances in presence of acid or basic catalysts. As hardening agent (to convert the products from the fusible into the infusible state) furfuramide is used, and this substance may also be used to harden the initial fusible product of a phenol–formaldehyde condensation. Synthetic resins may also be obtained without the use of a catalyst by heating furfuraldehyde–phenol mixtures to 215—230°—a much higher temperature than is ordinarily used in such processes. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 187,480.) S. S. WOOLF.

Production of resinous bodies. E. SCHAAL (E.P. 243,556, 20.1.25).—Copal substitutes are produced by heating glyceryl esters of resin acids (ester-gums) or rosin, mixed with oxidising and dehydrating agents, to a temperature below melting-point in a rotating drum to which a stream of air is admitted. A typical mixture is 100 pts. of finely-powdered ester-gum, 0·3 pt. of cobalt acetate (or manganese borate or resinate), and 40 pts. of anhydrous sodium sulphate (or 20 pts. each of the latter and dry sodium chloride). The temperature is raised from 30° to 100°, the stream of air inhibiting melting and caking. The mass is well washed with warm water and dried by heating to incipient fusion. A similar process serves for rosin, except that the temperature during the heat treatment must be maintained about 10° lower. The products are suitable for spirit- as well as oil-varnishes, but in the former case 5—10% of glycerin or a mixture of glycerin and linseed oil is added as plasticising agent. S. S. WOOLF.

Vulcanising fat substances. J. H. VAN DER MEULEN (U.S.P. 1,566,785, 22.12.25. Appl., 14.7.23).—See E.P. 201,531; B., 1924, 917.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Periodic tapping of *Hevea* [rubber trees]. I. Experimental basis of periodical tapping. J. GRANTHAM. **II. Effect of alternate periods of tapping and resting on the quality of the rubber.** B. J. EATON and R. O. BISHOP (Malay. Agric. J., 1925, 13, 342—350).—Alternating periods of daily tapping with periods of rest, the periods ranging from ½ to 2 months, and tapping on half the circumference of the tree, gives a yield of rubber at least equal to that of daily tapping on one-third of the circumference; tapping for alternate periods however causes fluctuations in the concentration of the latex. The rubber obtained immediately after a period of rest is slow in vulcanisation (cf. de Vries and Spoon; B., 1925, 17) but the effect of periods of rest of one month or less is greater than was observed by earlier workers. D. F. TWISS.

Aggregation and re-aggregation of raw rubber in the normal and deformed state. M. KRÖGER (Gummi-Ztg., 1926, 40, 782—784).—The effect of cold in producing a change in rubber comparable with that of ordinary vulcanisation has a parallel in the effect of pressure. Masticated or heated rubber on storage at the ordinary temperature tends to regain its strength by a process of "re-aggregation," the recovery being greater in "Para hard cure" rubber than in crêpe rubber. When rubber rings were stored for one month in a state of tension, the sample under 180% extension exhibited a greater final tensile strength than corresponding samples whether under 800% extension or unstretched, the influence of the various degrees of extension being analogous with that of progressive increase in the period of an ordinary vulcanisation process; the term "deformation-vulcanisation" is proposed for the effect of strain on the recovery of heated or masticated rubber. Stretched rubber is also attacked by bromine or iodine much more rapidly (per unit of surface) than the unstretched material; this greater reactivity of stretched rubber is of much significance in connexion with the resistance of rubber to atmospheric oxidation. D. F. TWISS.

Action of nitrogen tetroxide on caoutchouc. F. EMDEN (Ber., 1925, 58, 2522—2527).—If a solution of caoutchouc from *Hevea brasiliensis* or *Parthenium argentatum* in carbon tetrachloride is slowly added to a solution of nitrogen tetroxide in the same solvent, the nitrosate, $C_{10}H_{16}O_6N_2$, is precipitated in large, white flocks, decomp. 90—95°. The composition of the product is remarkably constant even when the temperature of the solution is varied between —15° and +5° and the duration of the reaction from 10 to 90 min.; the yield is approximately theoretical. The product is initially insoluble in acetone in which, however, it dissolves on prolonged contact, yielding a clear, reddish-brown solution from which it is precipitated in a brownish-yellow soluble form by addition of water. The latter variety is the more stable and does not evolve carbon dioxide or other gas when dissolved in boiling acetone. Its production appears to be due to transformation within the molecule. The formation of an oxidised product under conditions specially designed to exclude oxidation as far as possible

renders improbable the existence of the "dinitro-caoutchouc, $C_{10}H_{16}O_4N_2$" described by Weber (Ber., 1902, **35**, 1947) as derived from caoutchouc and nitrogen dioxide. H. WREN.

Comparative resilience of leather and rubber heels. WILSON.—See XV.

PATENTS.

Manufacture of new accelerators and of intermediate compounds for use in the vulcanisation of rubber. BRITISH DYESTUFFS CORP., C. T. CRONSHAW, and W. J. S. NAUNTON (E.P. 244,159, 9.9.24).—Diarylthiocarbamides containing an alkylamino-group in each aryl nucleus are desulphurised with formation of similar substituted diarylguanidines; thus tetramethyldiaminodiphenylthiocarbamide and diethyldiaminodi-*p*-tolylthiocarbamide are converted into tetramethyldiaminodiphenylguanidine and *p*-diethyldiamino-*m*-dimethyldiphenylguanidine respectively. The products have an increased accelerating action as compared with diarylguanidines, without danger of pre-vulcanisation. D. F. TWISS.

Vulcanising rubber. S. M. CADWELL, Assr. to NAUGATUCK CHEMICAL CO. (U.S.P. 1,564,824–5, 5.12.25. Appl., [A] 15.5.22, [B] 6.8.25).—Rubber is mixed with a vulcanising agent and (A) a thioaldehyde or (B) an arylated thioaldehyde, and vulcanised. D. F. TWISS.

Vulcanisation of caoutchouc. J. M. GILLET, Assr. to GOODYEAR TIRE AND RUBBER CO. (U.S.P. 1,566,247, 15.12.25. Appl., 31.7.22).—A fibrous material is produced by mixing a relatively small quantity of fibres and rubber in a rubber solvent, precipitating the rubber on the fibres, introducing sulphur dioxide and hydrogen sulphide into the liquid, straining the fibres from the liquid so as to obtain a felted mass, and then drying. D. F. TWISS.

Uniformly and intimately mixing materials with rubber latex. E. HOPKINSON (U.S.P. 1,567,506, 29.12.25. Appl., 16.10.22).—See E.P. 205,487; B., 1925, 18.

Alkali polysulphides (E.P. 243,394).—See VII.

Hard rubber varnishes (E.P. 243,966).—See XIII.

Waterproof paints (E.P. 244,150).—See XIII.

XV.—LEATHER; GLUE.

Astringency of vegetable tanning materials. E. CREDE (J. Amer. Leather Chem. Assoc., 1925, **20**, 573—576).—The Wilson–Kern method of tannin analysis (cf. B., 1921, 819 A) measures only the tans which when fixed by hide resist washing with water. Assuming that these correspond with the astringent tans, the astringency can be defined as the ratio, tannin by Wilson–Kern method : tannin by official method. The following figures have been obtained:—Quebracho 72·5%, hemlock 63·0%, cutch 64%, chestnut oak bark 53·1%, wattle bark 54%, chestnut wood 46·5%, valonia 37·5%, divi-divi 21·3%, myrobalans 16·5%. D. WOODROFFE.

Comparative resilience of leather and rubber heels. J. A. WILSON (J. Amer. Leather Chem. Assoc., 1925, **20**, 576—579).—Measurements of the rebound of a weight dropped on to various samples of leather and rubber heels showed that only one make of rubber heels had a resilience comparable with that of oak-tanned leather. D. WOODROFFE.

Preparation of leather for analysis. L. BALDERSTON (J. Amer. Leather Chem. Assoc., 1925, **20**, 583—589).—Samples of leather prepared by sawing and slicing respectively show differences in moisture content, in proportion of tans to non-tans in the water extract, and in fat content. The author has devised a machine which slices the leather, the knives being arranged similarly to those in an ensilage cutter, and another set of knives being arranged to slice the pieces longitudinally in order to subdivide them into small pieces. Sole leather in this cutter yields pieces $\frac{1}{4}$ in. $\times$ $\frac{1}{8}$ in. $\times$ $\frac{1}{100}$ in. D. WOODROFFE.

Machine for preparing leather samples for analysis. A. C. ORTHMANN (J. Amer. Leather Chem. Assoc., 1925, **20**, 579—583).—Detailed drawings are given of a machine for preparing any kind of leather samples for analysis. Lamson cuttee heads are used. D. WOODROFFE.

Reduction of gelatin. ABDERHALDEN and SCHWAB.—See A., 1926, 83.

PATENTS.

Stuffing of chrome-tanned heavy leather. R. H. PICKARD, D. JORDAN-LLOYD, and A. E. CAUNCE (E.P. 243,438, 27.8.24).—Chrome-tanned leather in the wet "blue" state is sprayed with, or steeped in, acetone until the water content of the leather is reduced to 14—20%, or until a sample cutting dried at 57° will wet back in water. The acetone is then removed by subjecting the leather to warm air at 57°, and the leather is dipped in or treated with a suitable stuffing material. D. WOODROFFE.

Manufacture of gelatin. A. H. TOD (E.P. 244,019, 8.8.25).—A continuous supply of hot water is passed slowly through a rotating basket containing gelatinous matter, and the liquid is removed at the same rate. The inlet and outlet pipes are fitted with valves which are connected together. The rate of flow is regulated so as to allow the liquid to reach the desired degree of concentration. The apparatus may or may not be operated in a total or partial vacuum. D. WOODROFFE.

Tanning skins and hides. J. HELL (U.S.P. 1,567,644, 29.12.25. Appl., 29.8.21).—See E.P. 180,758; B., 1922, 602 A.

XVI.—AGRICULTURE.

Irrigation and soil reaction. SCHWÖRER (Z. Pflanz. Düng., 1925, B4, 557—563).—The author discusses possible explanations of the fact that irrigated meadow land, in the Black Forest area, carries a heavier crop and a markedly better type of herbage than similar meadows which are not under irrigation. The soils are naturally acid; both the "actual" and the "exchange" acidity are much reduced where irrigation is practised. The irrigation water is very poor in bases, but such large quantities are used that the cumulative effect of the small percentage present probably eventually accounts, to a great extent, for the change in reaction. Further, an appreciable loss of iron from the irrigated soils can be demonstrated. Manures are seldom applied and it remains difficult to explain the continued good yields from these meadows. C. T. GIMINGHAM.

Neubauer method and determination of the nitrogen requirements of soils. E. BLANCK and F. SCHEFFER (Z. Pflanz. Düng., 1925, B4, 553—556).—An attempt to apply the Neubauer "seedling" method (cf. Z. Pflanz. Düng., 1923, A2, 329) to the determination of the nitrogen requirements of soils gave results which were not in agreement with those obtained by careful vegetation experiments. The method was applied exactly as it is used for the determination of the potassium and phosphorus requirements of soils, but it is recognised that, in dealing with nitrogen, a number of new factors are involved. The data are published as a contribution to the working out of the method and do not detract from its value in regard to potassium and phosphorus. C. T. GIMINGHAM.

Influence of manuring with straw on the yield [of various crops]. GERLACH (Z. Pflanz. Düng., 1925, B4, 534—550).—Details of field experiments are given which lead to the conclusion that the use of straw as manure is, in general, uneconomical. Under practical conditions, most of the nitrogen is washed out of the soil or lost as gas during the period when the crop cannot be there to make use of it. C. T. GIMINGHAM.

Physics of spray liquids. II. Protective action of various substances on lead arsenate suspensions. III. Ease of formation of emulsions. IV. Creaming capacity of emulsions—paraffin solutions. R. M. WOODMAN (J. Pom. Hortic. Sci., 1925, 4, 78—94, 95—103, 184—195; cf. B., 1924, 803).—The formation and stabilisation of suspensions of lead arsenate in water by various protective agents has been studied, using a method involving siphoning off fractions of the suspension at definite intervals and measuring the amount of lead arsenate present by density determinations. Gelatin at concentrations between 0·5% and 0·2% was the most efficient of the materials examined; calcium caseinate was almost as good in promoting the formation of suspensions, but was less effective as a stabiliser; dextrin and starch gave intermediate results; sodium caseinate and soap were ineffective, and the free alkali present tended to form soluble arsenates. The use of gelatin together with finer grinding of the lead arsenate than is usually adopted is recommended. The preparation of emulsions by intermittent shaking (cf. Briggs, B., 1920, 341 A) has been used to compare the effect of various conditions such as temperature, concentration of emulsifier and mixtures of emulsifiers on ease of emulsification. The "cream" which forms on the surface of emulsion mixtures is considered to be the only true emulsion, and a distinction is drawn between the true stability of an emulsion, *i.e.*, the resistance to separation into layers of the disperse and continuous phases, and the stability to creaming. Rapid creaming is usually due to large differences in the densities of the two phases; experiments are recorded on the addition of a third substance to equalise the densities and so make the mixture cream more slowly. Creaming of soap solution–paraffin oil emulsions is greatly retarded by addition of cresol, and in some cases, actual solution of considerable amounts of oil can be obtained. C. T. GIMINGHAM.

Influence of media on germination of seeds. CERIGHELLI.—See A., 1926, 99.

PATENTS.

Manufacturing solid calcium nitrate. BADISCHE ANILIN- U. SODA-FABR., Assees. of C. EYER and R. GRIESSBACH (U.S.P. 1,564,410, 8.12.25. Appl., 29.6.25).—Solid calcium nitrate containing a small quantity of an ammonium salt "in molecular distribution" is claimed. A. R. POWELL.

Simultaneous manufacture of ammonium chloride and sulphate, especially for fertilisers. H. DANNEEL (G.P. 417,214, 29.7.24).—A mixture of sulphur dioxide, chlorine, and ammonia in the stoichiometrical proportions is brought into contact with at least the requisite amount of water for the reaction. The product may be utilised directly as a fertiliser, or ammonium chloride and sulphate may be separated from the product by fractional crystallisation. A. R. POWELL.

Treating fertilisers. P. T. AXELSEN, Assr. to NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (U.S.P. 1,567,408, 29.12.25. Appl., 14.1.24).—See E.P. 210,409; B., 1925, 20.

Product [insecticide] resulting from a reaction between barium and a metal of the sulphur group. C. DICKENS (E.P. 244,951, 20.2.25).—See U.S.P. 1,536,379; B., 1925, 611.

Coating particles of copper sulphate (U.S.P. 1,562,821).—See VII.

XVIII.—FERMENTATION INDUSTRIES.

Amylase from germinated wheat and rye. N. M. TAYLOR, M. SPENCER, and M. HOUSE (J. Amer. Chem. Soc., 1925, 47, 3037—3039).—Wheat and rye were germinated for 12 hrs. in a nutrient solution (cf. Bakke and Erdmann, Amer. J. Botany,

1923, 10, 8), and then spread on paraffined cheese-cloth supported over the solution for 3 days at 16—18°, with access of air. The grain was then washed, air-dried, and ground to a fine flour. The latter was soaked for 3 hrs. in 2½ times its weight of cold water, dialysed in a collodion container for 24 hrs., filtered, the active material precipitated by adding ammonium sulphate, and purified by dialysis in aqueous solution till free from sulphate (cf. Sherman and Schlesinger, A., 1913, i, 1255; 1915, i, 183, 604). The solution was then concentrated to one-half its original volume by pervaporation (cf. Kober, A., 1917, ii, 295), this step being omitted in the case of the rye. Addition of 60% of cold alcohol precipitated inactive material, after removal of which further addition of alcohol precipitated the active amylase. The products thus obtained from wheat and rye gave typical protein tests, showed optimum activity at p_H 3·5—5·1, and compared favourably with amylase from malt (cf. Sherman, Thomas, and Baldwin, A., 1919, i, 181).

F. G. WILLSON.

See also A., 1926, 93, **Diastase** (SYNIEVSKI). 94, **Activation and heat stability of rennin and pepsin** (WOHLGEMUTH and SUGIHARA). 95, **Plant reductases** (PALLADIN, PLATISCHENSKI, and ELLADI); **Co-zymases in different fermentations** (VIRTANEN); **Azozymase and cozymase** (NEUBERG and GOTTSCHALK); **Action of adrenaline etc. on self-fermentation of yeast** (POPPER); **Influence of oxygen on alcoholic fermentation** (MEYERHOF). 96, **Influence of ions on sugar assimilation of oxygenated yeast** (LIEBEN and LÁSZLO); **Fixation of nitrogen by yeast** (FULMER and CHRISTENSEN); **Behaviour of acetylmethylcarbinol to yeast** (NEUBERG and KOBEL). 97, **Lactic acid formation by bacteria** (NEUBERG and GORR).

PATENTS.

Production of practically alcohol-free beverages. J. F. MEYER and H. LÜCKER (E.P. 244,988, 11.5.25).—See U.S.P. 1,537,252; B., 1925, 564.

Production of yeast. S. SAK, Assr. to FLEISCHMANN Co. (U.S.P. 1,566,431, 22.12.25. Appl., 14.12.20; renewed 7.11.25).—See E.P. 153,667; B., 1921, 57 A.

XIX.—FOODS.

Determination of coconut oil in margarine. S. H. BERTRAM, J. P. K. VAN DER STEUR, and F. VERHAGEN (Chem. Weekblad, 1925, 22, 549—550; cf. *ibid*, 1923, 20, 610).—The fat is hydrolysed in glycerin by potassium hydroxide; the solution is cooled and diluted, and the insoluble magnesium soaps are precipitated at 70—80° by addition of magnesium sulphate with continuous stirring. The solution is cooled to 20° and allowed to stand for 10 min., the soaps are filtered off, and the filtrate is neutralised and divided into two parts. One part is treated with sodium nitrate and silver nitrate, the insoluble silver soaps are filtered off after settling, and the excess of silver, A, determined in the filtrate by titration with thiocyanate. The second part is treated with a large excess of silver, the precipitated silver salts are filtered off, the filtrate is acidified, and the volatile acids are distilled off in the Polenske apparatus, and determined (B) by titration with sodium hydroxide. From the values A and B the percentages of butter and coconut fat are obtained by reference to a standard chart. S. I. LEVY.

Detection of coconut oil in cacao butter and in products containing the latter. J. HANUS and B. KOMOROUSOVÁ (Chem. Listy, 1925, 19, 394—397).—The ethyl ester values of coconut oil and of cacao butter differ by about 20 units and a determination of the ester value of a mixture will therefore give clear indications of the presence of each fat.

A. R. POWELL.

Determination of coconut oil in chocolate fondants. J. LUKAS (Chem. Listy, 1925, 19, 397—399).—The fats are extracted from the chocolate by means of ether and the ethyl ester value is determined. The value for cacao butter is 4 and for coconut oil 80; from these figures the proportion of each substance in a mixture of the two may be readily calculated if the value for the mixture is known. (Cf., B., 1925, 929; Vaubel, B., 1924, 524, and *supra*.) A. R. POWELL.

Detection of small quantities of arsenic in cocoa. H. LÜHRIG (Pharm. Zentr., 1926, 67, 1—3).—Owing to the difficulty of destroying the organic matter of cocoa without loss of arsenic; the usual methods of determining traces of the element in cocoa yield low results and require considerable time. As little as 0·024 mg. of arsenic may be detected in 15 g. of cocoa by boiling the powder with 75 c.c. of 16% hydrochloric acid and a bright strip of copper for 20—25 min. A grey to black deposit which is converted into white octahedral crystals on gently heating over a small flame indicates the presence of arsenic. A quantitative analysis may then be conducted by evaporating to dryness a mixture of 15 g. of the cocoa powder with 10 c.c. of water, 0·5 g. of magnesia, and 5 c.c. of nitric acid, heating the residue until it carbonises, extracting the soluble matter with hydrochloric acid, evaporating the solution with sulphuric acid, and carrying out the Marsh test. A. R. POWELL.

Photoactivation of vitamin-A etc. by ultraviolet light. HAMANO—See A., 1926, 98.

Vitamin-B. LEVENE and VAN DER HOVEN.—See A., 1926, 98.

Nitrogenous constituents of lucerne. VICKERY.—See A., 1926, 99.

PATENTS.

Process for adding A-vitamins to oils. AARHUS OLIEFABRIK A./S., and K. H. HANSEN (E.P. 243,907, 30.12.24).—Vegetable, animal, fish, or edible mineral oils are agitated with an aqueous solution of soap, formed by saponifying a vitamin-containing marine animal oil, such as cod-liver oil,

with soda or potash lye. During the agitation, preferably with exclusion of air, the A-vitamins present in the marine animal oil in the form of unsaponifiable emulsified particles are dissolved by the oils under treatment. It is beneficial to add to the soap solution about 0·5—1% of a fat solvent such as benzine. H. M. Langton.

Apparatus for treating the vapours arising from plants for drying wet organic matter. J. R. Sterling (E.P. 243,780, 6.8.24).—In the production of animal foods etc. by treating wet organic matter, such as meat, bones, fish, or vegetable substances with a hot gas, such as steam, in a closed container connected with a jet or barometric condenser, offensive vapours are deodorised by means of deodorants, such as permanganate, creosote solutions, etc. supplied to the condenser with the cooling liquid. H. M. Langton.

Preparing fruit juice. W. C. Pierce (E.P. 237,592, 13.7.25. Conv., 24.7.24).—See U.S.P. 1,556,572; B., 1925, 968.

Vacuum evaporating apparatus (U.S.P. 1,560,598).—See I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

See A., 1926, 81, **Synthesis of galegine** (Späth and Spitzy). 82, **Alkaloid of *Cocculus laurifolius*** (Kondo and Kondo); **Feebly toxic alkaloidal derivatives** (Polonovski and Polonovski); **Oxyberberine, palmatine, and tetrahydrojatrorhizine** (Späth and Quietensky); **Oxyacanthine** (Späth and Kolbe). 83, **Codeine and thebaine** (Gulland and Robinson). 91, **Physiological activity of benzyl compounds** (Jensen). 97, **Chemical properties of insulin** (Scott); **Chemical composition of insulin** (Glaser and Halpern). 98, **Dialysis and adsorption of insulin** (Dingemanse); **Stability of insulin** (Dingemanse). 99, **Alkaloid content of *Lupinus luteus* during growth** (Sabalitschka and Jungermann).

Methods of washing gases. Weissenberger, Henke, and Sperling.—See I.

Patents.

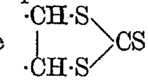
Manufacture of esters of unsaturated acids. O. Y. Imray. From Soc. of Chem. Ind. in Basle (E.P. 243,510, 7.11.24).—Therapeutically active esters of unsaturated acids are prepared by esterifying with cholesterol an aliphatic or aliphatic-aromatic acid having a triple linkage, or an aliphatic acid having at least one double linkage and low in the series, or an aliphatic-aromatic acid having at least one double linkage in the side chain. Their therapeutic activity is enhanced by using them (externally or injected) in solution in phenylacetylene and camphor. The preparation of the *cholesteryl esters* of phenylpropiolic acid (m.p. 153·5° with change of colour after softening at 148°), crotonic acid (m.p. 149° after softening at 90°), tetrolic acid (softens at 115°, melts at 120° to a turbid and at 124° to a clear liquid), and α-benzylidenepropionic acid (softens at 103°, melts at 106° to a turbid and at 175° to a clear liquid, is described. W. T. K. Braunholtz.

Manufacture of *N*-methylsulphites of secondary aromatic-aliphatic amines. Farbw. vorm. Meister, Lucius, u. Brüning, M. Bockmühl, and A. Schwarz (E.P. 243,925, 24.1.25. Addn. to 164,002; cf. U.S.P. 1,426,348; B., 1922, 786 a).—In preparing therapeutically active *N*-methylsulphites of secondary aromatic-aliphatic amines (other than methyl-, ethyl-, or benzyl-aniline, an *N*-alkylated anthranilic acid or its nucleus-substitution product) the formaldehyde–bisulphite compound may be replaced by formaldehyde and a bisulphite separately, added in any order, or by treating a condensation product from a primary aromatic amine and formaldehyde with an alkylating agent and then with a bisulphite. Details are given of various methods of preparing the methylsulphites of *N*-methyl-*p*-phenetidine, and 1-phenyl-2:3-dimethyl-4-methyl (or ethyl)-amino-5-pyrazolone. W. T. K. Braunholtz.

Preparation of alkyl esters of 2-quinolone-4-carboxylic acid. E. Thielepape (G.P. 416,769, 3.11.21; cf. A., 1922, i, 271).—*N*-Aryl- or *N*-aryl-*N*-alkylacetamides are treated with alkyl oxalates and sodium ethoxide, the resulting compounds of structure $R.N.R^1.CO{\cdot}CH_2{\cdot}CO{\cdot}CO_2R^2$ (R=aryl, R^1=H or alkyl, R^2=alkyl) being treated with a dehydrating agent, *e.g.*, concentrated sulphuric acid. B. Fullman.

Preparing esters of carbithionic acids and thiocarboxylic acids of the pyrazolone series. Chem. Fabr. auf Actien vorm. E. Schering (G.P. 416,860, 17.2.24).—Pyrazolones are treated in carbon disulphide with aluminium chloride and alkyl chloroformates or alkyl halides, and the esters formed hydrolysed. For example, to 1-phenyl-2:3-dimethyl-5-pyrazolone and ethyl chloroformate or ethyl bromide, in boiling carbon disulphide, aluminium chloride is added in two portions during 3 hrs., and the solution boiled for 3 hrs. On removing the solvent in a vacuum and treating the residue with ice-water there is obtained *ethyl 1-phenyl-2:3-dimethyl-5-pyrazolone-4-carbithionate*, m.p. 178·5—179°, which on hydrolysis with water or alcoholic potassium hydroxide gives the corresponding *thiocarboxylic acid*, m.p. 81—82°. *Ethyl 1-phenyl-5-methyl-3-pyrazolone-4-carbithionate*, m.p. 114° and the *carbithionic esters* of 1-phenyl-3-methyl-5-pyrazolone, m.p. 81—82°, of 3-methyl-5-pyrazolone, m.p. 186°, and of 5-pyrazolone, m.p. 184—185°, are described. The compounds have therapeutic application. B. Fullman.

Preparation of organic thiocarbonates. C. Guerry (F.P. 563,214, 20.5.22).—Aqueous solutions of the alkali salts of organic acids containing an olefinic linkage (especially unsaturated fatty acids of formula $C_nH_{2n-2}O_2$) such as oleic, ricinoleic, and linoleic acids, and resins and colophony (abietic acid) are treated with a solution of sulphur in

carbon disulphide approximating in composition to CS_3. A ring of the annexed structure  is formed at the double bond, the products being *derivatives* of trithiocarbonic acid. They dissolve animal or vegetable fats, oils, waxes, resins, naphthene hydrocarbons, aromatic hydrocarbons and their substitution products, and spermaceti. They are unstable in presence of mineral acids. They are used in degumming silk; for dissolving fats to produce leather dressings; and as fungicides and insecticides, *e.g.*, as plant sprays in the form of their saturated solutions in coal-tar oil or in aromatic hydrocarbons or their nitro-derivatives.

B. FULLMAN.

Activated carbon (E.P. 243,801).—See II.

Alkali polysulphides (G.P. 243,394).—See VII

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Dark-room infection by red-sensitising agents. E. von Angerer (Physikal. Z., 1925, **26**, 864).—Ordinary photographic plates after development and fixation frequently show a set of fine wave-like black streaks which disfigure and sometimes ruin the negatives. The effect was traced to the action of traces of Pinacyanol and other red-sensitising agents, which are effective at very great dilution. Sufficient Pinacyanol is retained in a washed developing dish to affect the next plate to be developed in red light, using an alkaline developer. The streaks can best be avoided by using separate dishes for panchromatic or sensitised plates, or else by cleaning with chromic acid after using Pinacyanol.

R. A. MORTON.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Blasting-powder composition. C. D. PRATT, Assr. to ATLAS POWDER Co. (U.S.P. 1,563,924–6, 1.12.25. Appl., 31.5.24).—Blasting-powder compositions contain the constituents of black blasting powder, together with (A) 5—35% of carbohydrates and 1—15% of a metal chloride, (B) 5—35% of one or more carbohydrates and 1—15% of one or more tartrates and oxalates, or (C) 1—35% of carbohydrates and other non-explosive organic material deficient in oxygen. L. A. COLES.

Manufacture of propellent powders. C. R. FRANKLIN (U.S.P. 1,564,549, 8.12.25. Appl., 19.12.23).—Nitroglycerin is added with agitation to a mixture of nitrocellulose (N 12·60% approx.) with a large excess of water. The excess of water is removed and a stabiliser and flash-reducing compound are added. After mixing, the material is passed through heated pressure-rolls, seasoned, treated with a volatile solvent, passed through hot rolls, cut, and dried. S. BINNING.

Manufacture of detonators and cartridges. H. W. BROWNSDON, and NOBEL'S EXPLOSIVES Co. (E.P. 243,771, 10.6.24).—Detonator shells made of an aluminium–silicon alloy, in which the content of silicon is from 2 to 5% are loaded with an azide, *e.g.*, lead azide, either alone or in admixture or conjunction with other suitable substances.

S. BINNING.

Explosive. N. A. UNGER (U.S.P. 1,566,784, 22.12.25. Appl., 14.3.24).—See E.P. 220,619; B., 1925, 268.

Reducing viscosity of nitrocellulose (U.S.P. 1,564,689).—See V.

Removing pyridine from nitrocellulose (U.S.P. 1,564,765).—See V.

XXIII.—SANITATION; WATER PURIFICATION.

Composition of mud balls [from water filters]. W. S. MAHLIE (J. Amer. Water Works Assoc., 1925, **14**, 420—422).—No relation between the size and the composition of mud balls was established by the tests made on balls of different sizes taken from a filter which had received water coagulated with iron for nearly 5 years and then water coagulated with alum for nearly 6 months. The moisture contents, determined by drying in open pans at the ordinary temperature, varied from 27 to 42%. Washing losses, which were determined by stirring 1000 g. of the sample with water, allowing the sand to settle, pouring off the supernatant liquid, and repeating the process until the washings came away clear, varied from 16·8 to 22·8%. An air-dried evaporation residue from the washings showed upon analysis: ignition loss 31·61%, SiO_2 30·00%, Fe_2O_3 + Al_2O_3 29·16%, CaO 7·92%, MgO 2·35%, Mn trace. Observations made indicated that anaerobic bacterial action was taking place within the centre of the balls.

W. T. LOCKETT.

Iron incrustation in water pipes. D. ELLIS (Water and Water Eng., 1925, **27**, 352—354).—The nodules of a hard incrustation which appeared in cast iron pipes coated with tar appeared to be completely separated from the iron of the pipe by the tar coating, and as they were found to contain iron bacteria, their formation was at first ascribed to deposition of iron from the water, which contained 0·04 p.p.m. of iron, and 10,000—60,000 bacteria per c.c. Experiments showed, however, that formation of the incrustation proceeded independently of the bacteria, and examination of the tar coating showed it to be porous to water, and it was concluded that the incrustation was derived from the pipe and not from the water. The water had p_H 6 and contained a comparatively large amount of organic matter (8·7 p.p.m. oxygen absorbed). Ferruginous festoons and streamers which form in water pipes appear to be a purely biological problem, while discoloration of water due to iron in colloidal solution is brought about by both physical and chemical factors. R. E. THOMPSON.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 5, 1926.

I.—GENERAL; PLANT; MACHINERY.

Adsorption of gases [ammonia, sulphur dioxide, chlorine] by wood charcoal at pressures exceeding 1 atm. F. A. Henglein and M. Grzenkovski (Z. angew. Chem., 1925, **38**, 1186—1188). —The apparatus comprises essentially two glass limbs, one of which contains the charcoal and is evacuated and sealed, whilst the other is graduated and contains the liquefied gas. When a protruding neck of the charcoal chamber is broken, connexion is established between the limbs, and some of the liquefied gas volatilises and is adsorbed by the charcoal, the quantity adsorbed being calculated from the loss in volume of the liquefied gas. With ammonia and at a temperature of 30°, the charcoal used reached saturation at 8·19 atm., and then contained 34% of its weight of gas. With sulphur dioxide the same charcoal showed no saturation point, and at 25° and 3·35 atm. had taken up 62% of its weight of gas. The results with chlorine were less regular, and some chemical action of the gas on the charcoal is assumed. At 15° and 4 atm. the charcoal took up about 70% of its weight of chlorine within a few minutes. W. T. K. Braunholtz.

Application of rotating tubes in crystallisation and evaporation. H. Barkholt (Chem.-Ztg., 1926, **50**, 45—46).—The advantages of the apparatus described (cf. G.P. 333,304 and 351,947; B., 1921, 422 A; 1922, 737 A) lie in their continuous operation, low maintenance and installation costs with respect to the yield, immunity from corrosion and consequent purity of the product, and wide range of applicability. Since the evaporator works on the same principle as the crystalliser, the two operations may be performed in one tube, the evaporating portion of which is heated by flue-gases or other heating agent. E. S. Kreis.

Laboratory kiln for obtaining high temperatures. F. H. Norton (J. Amer. Ceram. Soc., 1925, **8**, 826—828).—A small cubical chamber has a tuyère (through which gases of combustion enter from a Premix blower) placed near the crown, so that the flames pass over the charge and down to flues in the bottom, from which the gases pass out through four stacks, one at each corner of the kiln. The efficiency of the furnace is due to careful design of the tuyère, the combustion space, the baffles, and the flues, and not to preheating or to heavy insulation. A temperature of 1400° is reached in one hr. and 1800° in 4 hrs. The actual flame temperatures are estimated to be almost as high as the maximum theoretical values for the natural gas used in the experiments. F. Salt.

Technical dispersoid analysis. F. V. von Hahn (Kolloid-Z., 1925, **37**, 377—379).—A discussion of the measurement of the degree of dispersion by methods specially applicable to the control of industrial processes, namely methods depending mainly on filtration, ultrafiltration, microscopy, and sedimentation. N. H. Hartshorne.

Plasticity. A. de Waele (Kolloid-Z., 1925, **38**, 27).—The extrusion of an heterogeneous system through a capillary orifice under pressure comprises a combination of two régimes, *i.e.*, that of the shear of the continuous phase resulting in a velocity gradient within it, and mere extrusion of accompanying unshearable disperse phase not susceptible to a velocity gradient. By deriving the empirically-obtained proximate equation for the "shear" of a heterogeneous system through a capillary from these principles, ψ in the equation P/V^{ψ}=constant, is shown to denote the volume proportion of shearable, truly viscous phase. Many, if not all heterogeneous systems show in addition, evidence of a static elasticity or yield value (f), the actual resultant of which is, however, variable in magnitude with the stress applied, thus: yield value at any moment $(F)=fe^{-\text{stress}}$, where e=log base. This static yield value is recoverable on rest according to the relationship: $F=fe^{-\eta/t}$. The complete equation showing the discontinuity in capillary shear owing to loss of yield value with stress then is:

$$\pi g R^4(P-fe^{-PR/2l})/8V^{\psi}l=\eta$$

The mechanism of this deflocculation on shear and re-flocculation with subsequent rest is suggested as being de-orientation and re-orientation respectively of molecules of the viscous phase at the boundary surfaces of the unshearable phase. A. de Waele.

Corrosion and erosion of steam turbine blading. E. Honegger (Brown Boveri Rev., 1924, **11**, 263—268; Chem. Abstr., 1925, **19**, 3471).—Corrosion and erosion usually assist one another, the latter exposing fresh surfaces to the action of the former. The incidence of both factors on the low- and high-pressure parts of steam turbine blading during operation has been examined, and the resistance of "stainless" steels has been studied. It appears that below a critical steam speed for any metal no erosion can take place. A. A. Eldridge.

Action of ammoniacal water on feed pipes and boiler plates. M. Tilgner (Chem.-Ztg., 1926, **50**, 48).—Water containing ammonia to the extent of 8—15 mg. per litre has been observed to produce heavy corrosion. E. S. Kreis.

Evaporation of mineral oils and its bearing on the use of oil-sprinkled air-filters. W. ALLNER (Z. angew. Chem., 1926, 39, 16—20).—The harmful consequences of the presence of oil in the supposedly purified atmosphere of electrical power-houses fitted with air filters of the Delbag-Viscin type (consisting of cells filled with oil-coated rings which retain dirt etc.) have been wrongly attributed to the use of the oil in the filter. Experiments are described showing that under the working conditions the volatilisation of oil is negligible, whilst formation of oil mist or mechanical entrainment of droplets is impossible. S. S. WOOLF.

See also A., 1926, 118, **Theory of atmolysis** (SAMESHIMA). 119, **Influence of thin surface films on evaporation of water** (RIDEAL). 122, **Determination of size of colloidal particles** (BURTON and REID); **Diffusion analysis** (AUERBACH); **Measurement of particle size** (KUHN). 124, **Theory of peptisation** (SEN). 142, **Viscosimetry** (STAUF).

Method of measuring porosity. ROBINSON.—See VIII.

PATENTS.

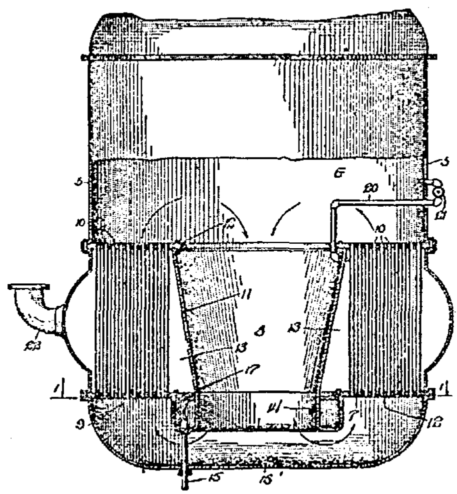
Manufacture of large [or single] crystals. GEN. ELECTRIC CO., Assces. of PATENT TREUHAND GES. FÜR ELEKTRISCHE GLÜHLAMPEN M.B.H. (E.P. 234,449, 2.3.25. Conv., 26.5.24).—The material to be converted into a single crystal is melted in a tube of which the largest diameter corresponds to the required thickness of the crystal and which is tapered to a smaller diameter and finally brought to a point at one end. The tube and its contents are then slowly cooled progressively along its length, starting at the small end. The material in the thin stem of the tube first solidifies to a single crystal before the melt in the main portion of the tube has solidified. As the tube is progressively cooled the crystal grows at the expense of the solidifying material. B. M. VENABLES.

Pulverising and drying of materials. O. Y. IMRAY. From INTERNAT. COMBUSTION ENGINEERING CORP. (E.P. 244,575, 28.11.24 and 21.4.25).—A pulverising mill of the type having a reservoir of material which is kept in agitation just before entry or re-entry into the actual grinding chamber (*e.g.*, the Raymond pulveriser), is operated with the bulk of the air travelling continuously in a circuit, but to effect drying as well as pulverising, a portion of the air saturated with moisture is withdrawn from the return conduit after leaving the collector for pulverised material and replaced by fresh air, which may with advantage be heated, and preferably enters the same return conduit at a point close to that of withdrawal. To prevent waste or dust in the withdrawn air it may be used to transport the finished material to the points of use, *e.g.*, to the furnaces in the case of powdered fuel. B. M. VENABLES.

Refrigeration. B. H. COFFEY (U.S.P. 1,565,795, 15.12.25. Appl., 9.8.20).—The pressure on a liquid refrigerant is gradually reduced to vaporise a portion without substantial heat exchange, and a further portion is vaporised at low pressure and temperature to produce the refrigeration. The pressure on the resulting mixture of liquid and vapour is then gradually increased, thus condensing part of the vapour without substantial heat exchange, and the remaining vapour is condensed at the higher pressure and a higher temperature. H. HOLMES.

Filter. W. P. COTTRELL (U.S.P. 1,565,988, 15.12.25. Appl., 28.10.24).—A compressible filtering medium is packed between spaced walls of impermeable material provided with perforations for passage of fluid. The perforations are arranged out of register to compel the fluid to follow a lengthy path between the walls, and means are provided for adjusting the pressure of the walls on the filtering medium. H. HOLMES.

Evaporator. M. J. KERMER (U.S.P. 1,566,539, 22.12.25. Appl., 15.8.23).—A "deck" or nest of tubes, 10, is situated within an evaporating chamber,



and a steam chamber, 13, surrounds the tubes. A trough, 14, is formed inside the steam chamber below the level of the tubes and water is forcibly projected through the trough, which is completely open to the steam. B. M. VENABLES.

Preventing loss by evaporation from storage tanks. R. E. WILSON, Assr. to STANDARD OIL CO. (U.S.P. 1,566,944, 22.12.25. Appl., 30.1.24).—A storage tank for volatile liquids is vented through a mass of silica gel, the temperature of which is regulated to be at least as high while the air is being drawn into the tank as the temperature prevailing during expiration. B. M. VENABLES.

Continuous drying kiln and method of drying ware. T. LARSSON, Assr. to NORTON CO. (U.S.P. 1,567,023, 22.12.25. Appl., 30.10.20).—A continuous drying kiln is provided with a series of heaters arranged along its length, each being thermostatically and separately controlled. Fans are provided to

circulate the air continuously across the kiln, and the air is also circulated longitudinally of the kiln, the return passage for this circulation being external to the actual drying chamber. The moisture content of the air is maintained constant by withdrawing a portion of the moist air and admitting a corresponding quantity of fresh air to the return passage. The amounts of the forward or longitudinal circulation and of air withdrawn for re-conditioning are also thermostatically controlled.
B. M. Venables.

Treatment of gases at high temperatures. M. Fourment (F.P. 593,298, 22.12.24).—Gases are passed through a chamber constructed of electrical-conducting material, which is brought to a high, uniform temperature by means of a high-frequency induction current. L. A. Coles.

Production of highly efficient catalysts. K. Wolf (G.P. 418,724, 30.7.21).—Insoluble, porous material is impregnated with solutions containing salts of catalytic metals and colloidal substances, and, after drying at low but slowly rising temperature, is heated at dull redness in the presence of reducing or oxidising agents, according to whether the metals or their oxides are required.
L. A. Coles.

[Platinum-ruthenium] catalyst. Ges. für nautische Instrumente G.m.b.H., and O. Martienssen (G.P. 418,868, 11.10.24).—Alloys of platinum and ruthenium are used instead of platinum as catalysts. L. A. Coles.

Ammonia-absorption refrigerator. C. Senssenbrenner (U.S.P. 1,568,476, 5.1.26. Appl., 16.12.24).—See E.P. 236,101; B., 1925, 656.

Refrigerating system. W. H. Carrier, Assr. to Carrier Engineering Corp. (U.S.P. 1,569,214, 12.1.26. Appl., 2.9.22).—See E.P. 230,936; B., 1925, 385.

Method of heating material at successively different temperatures. A. McD. Duckham and J. S. Morgan, Assrs. to Thermal Industrial and Chemical (T.I.C.) Research Co., Ltd. (U.S.P. 1,568,886, 5.1.26. Appl., 9.8.21).—See E.P. 174,690; B., 1922, 205 A.

Heat exchangers. C. Roszak (E.P. 235,149, 7.4.25. Conv., 5.6.24).

Rotary drying or torrefying apparatus. P. Scrive (E.P. 238,871, 7.8.25. Conv., 20.8.24).

Doll-head bearings for drying cylinders. S. C. Bullen (E.P. 245,548, 31.10.24).

Rotary dryer for whinstone, limestone, sand, clinker, and like material. R. H. and F. Kirkup and J. Thompson (E.P. 245,561, 1.8.25).

Apparatus for separating dust from air. J. B. Taylor (E.P. 245,243, 10.11.24).

Preventing and dissolving scale in steam boilers. P. Schenitza (E.P. 245,365, 10.7.25).

Furnaces. Vickers and Internat. Combustion Engineering, Ltd., and F. H. Rosencrants (E.P. 245,496, 6.10.24).

Washing apparatus for granular material. H. J. Greaves (E.P. 245,630, 24.2.25).

Combined evaporator and feed heating systems. Babcock and Wilcox, Ltd., and A. Spyer (E.P. 245,664, 16.6.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Conversion of ligneous plant substances into coal. III. Formation of sugars as an intermediate phase of the production of coal. C. G. Schwalbe and R. Schepp (Ber., 1925, 58, 2500—2502; cf. B., 1924, 240).—Humic acids are generally regarded as the precursors of coal and are possibly produced from sugars formed by hydrolysis. Experimentally it is shown that wood cellulose yields sugars when heated with 2% of oxalic acid at 170°. Dilute sucrose solutions when heated with oxalic acid at 130° afford humic acids which are transformed by concentrated solutions of magnesium chloride at 180° into substances resembling but not identical with lignite or coal. The production of the latter appears to depend on the simultaneous presence of lignin. When, therefore, spruce wood is heated successively with oxalic acid and magnesium chloride solutions a product is obtained which appears to have properties intermediate between those of lignite and coal. The carbon content increases with increasing duration of the treatment with magnesium chloride. H. Wren.

Heat of adsorption of gases by coal and charcoal. A. G. R. Whitehouse (J.S.C.I., 1926, 45, 13—20 T).—The heat of adsorption of carbon dioxide, sulphur dioxide, methane, oxygen, nitrogen, and carbon monoxide by charcoal and different types of coal at 0° was measured with a Bunsen ice-calorimeter. The values obtained for the heat evolved per c.c. of gas adsorbed are as follows:—Carbon dioxide 0·346—0·255 cal., sulphur dioxide 0·627—0·359, methane 0·245—0·199, oxygen 0·19—0·17, nitrogen 0·22—0·17, and carbon monoxide 0·198—0·188 cal. A simple exponential equation connecting the total heat evolved per gram of adsorbent with the total volume of gas adsorbed per gram of adsorbent is found to hold over the range covered by the experiments. The gases studied are adsorbed more slowly by coal than by charcoal, the difference being most pronounced in the cases of methane and nitrogen.

Refractories for gas retorts, with special reference to silica. W. Emery (Trans. Ceram. Soc., 1924–5, 24, 361—402; cf. B., 1924, 674; also Robinson, p.158)—An examination of the actual results with silica retorts in practice shows that in general

the throughput and rate of carbonisation are increased for horizontal retorts, this being chiefly due to the greater diffusivity of silica compared with fireclay at temperatures above 1000°, giving higher thermal conductivity. Silica retorts retain their rigidity and shape much better at higher temperatures than fireclay, the type of retort and setting, especially the position of the cross walls, exerting a considerable influence. The tendency to form less scurf on silica has also a favourable effect. Silica is more resistant to abrasion and to corrosion by salty coals or by ash in the producer gas. The high thermal expansion of silica can be dealt with by special design of the settings, and spalling troubles have been reduced by more careful attention to the manufacture of the silica. Experience to date seems to show that the advantages of the silica retort outweigh the disadvantages of higher capital outlay and the necessity for careful handling, and special precautions during the setting of the retorts. B. W. CLARKE.

Sodium chloride solution as a confining liquid for gas analysis. F. G. HOFFMANN (Z. angew. Chem., 1926, **39**, 23—24).—The solubility of carbon dioxide in dilute hydrochloric acid and in dilute sulphuric acid is almost as great as in pure water. The solubility in aqueous sodium chloride solution is not altered by addition of sulphuric acid, and the solubility of sodium chloride in dilute hydrochloric acid solutions is higher than in aqueous solutions of the same concentration. Thus the use of dilute acids or dilute acid solutions of sodium chloride as confining liquids offers no advantage over that of aqueous solutions. A 22% solution of chemically pure sodium chloride is recommended as the best confining liquid. In this solution carbon dioxide is less soluble than in one of commercial salt of the same concentration in the proportion 14 : 15.

E. H. SHARPLES.

Detection and determination of traces of carbon disulphide in small gas volumes. W. J. HUFF (J. Amer. Chem. Soc., 1926, **48**, 81—87).—The method of Harding and Doran (B., 1907, 1190) has been modified as follows:—A volume of gas, *e.g.*, coal gas or carburetted water-gas, containing at least 1 mg. of carbon disulphide is freed from carbon dioxide and moisture, and passed into a saturated solution of potassium hydroxide in absolute alcohol. The solution is then made acid with glacial acetic acid, excess of standard 0·01*N*-copper sulphate solution added, and the mixture left overnight. The copper xanthate, precipitation of which is aided by the presence of excess cupric ion, low acidity, and keeping the volume of solution as small as possible, is filtered off, and the excess of copper determined iodometrically. A sensitive qualitative method of detection is based on the same process.

R. CUTHILL.

Determination of water in mineral oils. S. REINER (Elektrotechn. Z., 1925, **46**, 1447—1448; Chem. Zentr., 1925, II., 2334).—For the determination of small quantities of water in mineral oils the oil is heated at 120° for 3 hrs. in a flask while a current of compressed air is passed through. The air is previously dried by passing it through a tower filled with calcium chloride, then through two U-tubes packed with phosphorus pentoxide, and the moisture removed from the oil is collected in two small U-tubes containing phosphorus pentoxide, protected from the atmosphere at the far end by a washing flask containing strong sulphuric acid.

A. R. POWELL.

See also A., Feb., 1926, 116, **Heat of combustion of salicylic acid** (BERNER); **Heats of combustion of standard substances** (SWIENTOSLAWSKI).

Evaporation of mineral oils. ALLNER.—See I.

Use of silica refractories. ROBINSON.—See VIII.

PATENTS.

Apparatus for drying or preheating coal or like material. S. R. ILLINGWORTH, and ILLINGWORTH CARBONIZATION Co. (E.P. 244,505, 9.9.24).—The coal or other material to be dried or preheated passes through vertical metal retorts, built up of tubes, or of plates which may be of H-section. These are enclosed in a retort setting of metal or refractory material, and can be heated by radiant heat from flue-tubes through which circulate hot gases from other processes, or from gas burners placed in the flue-tube itself. Each flue-tube forms a separate, easily manipulated unit. The retorts are stepped to provide vents for the escape of steam or gases into the space between the retorts and flue-tubes; or these gases may be taken away through perforated tubes passing down inside the retorts. Dampers are provided whereby, if required, the flue-gases may be caused to flow from any flue-tube to the interior of the retort setting and thence to the chimney. Scrapers, which can be reciprocated in the retorts, are provided for dealing with coal of high moisture content. The retorts are fed from bunkers having outlets of smaller size than the retorts, and agitating arms which may be moved to and fro in the outlets. The discharge of material from the retorts is controlled by reciprocating plates. Coal can be dried in this apparatus without coming into contact with the heating gases, whereby oxidation is reduced to a minimum. A. B. MANNING.

Binder [for fuel briquettes]. S. W. CARPENTER and G. N. WHITE (E.P. 244,517, 16.9.24; cf. Goodwin and White, B., 1926, 34).—Soft vegetable tissue, *e.g.*, straw, waste of potato, beet, or other crops, hemp waste, etc., is subjected to aerobic fermentation so that the whole of the non-gaseous products are retained in the mass, which is boiled with an alkali to form an agglutinous material. The product is used either alone, or dried and mixed with water, as a binder in the manufacture of fuel briquettes. The amount of binder required decreases with increasing briquetting pressure and with increased fineness of grinding of the fuel; from 4 to 8% is sufficient when using a pressure of 1—3 tons per sq. in. B. W. CLARKE.

Electrical [blast-furnace] gas purification. Lodge-Cottrell, Ltd. From Metallbank u. Metallurgische Ges. (E.P. 244,372, 29.8.25. Addn. to 177,117 and 238,480; B., 1923, 1 A; 1925, 790).—The gas after being pre-cleaned electrically and cooled by washing with water, is delivered directly, without preheating, into the apparatus for precipitating the fine dust particles electrically. H. Holmes.

Treatment of gas liquor. Recovery of phenols from ammoniacal liquor. L. R. W. Heffner (U.S.P. 1,566,795–6, 22.12.25. Appl., 16.12.24).—(A) Phenol is recovered from gas liquor by subjecting the liquor to a temperature of 98° or higher, in the presence of sufficient ammonia to drive off the phenol as phenoxide. (B) The liquor is distilled for the recovery of ammonia, the temperature of the liquor and of the vapours at the outlet of the still being kept at about 98° or higher, in order to drive off ammonium phenoxide with the ammonia. A. B. Manning.

Recovering benzene from gases. É. E.. M Fréchou (F.P. 594,531, 15.5.24).—Oils containing benzene are distilled in a vacuum and the vapours condensed at different temperatures. A. Geake.

Recovering by-products of coal distillation. É. M. E. Fréchou (F.P. 594,012, 3.5.24).—Activated charcoal is used for absorption under pressure. A. Geake.

Oil-shale retort. J. T. Pope (U.S.P. 1,564,271, 8.12.25. Appl., 24.10.22. Renewed 29.6.25).—The retort consists of a sheet metal casing mounted above a furnace with interposed firebrick slabs to prevent direct heating. An inclined table, capable of small longitudinal movement, is suspended in the metal casing and conveys crushed shale from one end of the retort to the other, entry and exit of the shale being controlled by roller gates. Superheated steam is distributed along the surface of the shale and a vapour offtake is provided. T. A. Smith.

Obtaining oil from oil sands. T. W. Pritchard (Can. P. 244,540, 27.2.24).—The sand is heated in a retort to a temperature between 340° and 430°, the oil vapours and gases are passed through a condenser, and a part of the uncondensed gas is led back through the retort in order to prevent the temperature rising to a height at which cracking of the oil occurs. A. B. Manning.

Obtaining oils from oil shales. Jura Oelschiefer-Werke A.-G. (G.P. 415,847, 20.8.20).—The shale is distilled by the direct passage through it of the hot gases from the combustion of the shale residue. The distillation of the shale and the combustion of the residue take place in separate chambers, one above the other, the material passing through the chambers in succession. A. B. Manning.

Purification of liquid fuels, in particular those containing alcohol. Soc. de Recherches et de Perfectionnements Ind. (F.P. 589,712, 16.1.24).—The liquid fuel is distilled in the presence of less than 1% of a purifying agent (metal, oxide, hydroxide, carbonate, alkyloxide, or carbide of an alkali or an alkaline-earth, or a mixture of these). A. B. Manning.

Motor fuel. J. D. Riedel A.-G. (G.P. 416,838, 16.8.21. Addn. to 414,245; cf. E.P. 202,805, B., 1923, 1012 A).—To the mixture of hydrogenated naphthalenes and low-boiling aliphatic hydrocarbons a further addition of a small quantity of ether or of an alcohol of higher boiling point is made so that a homogeneous mixture resistant to cold is produced. This prevents carbon formation in the cylinder when too rich a mixture is used, and at the same time gives an increase of power. A. B. Manning.

Catalyst and adsorbent for the production of hydrocarbons. Le Pétrole Synthetique (F.P. 589,831, 28.11.24. Conv., 20.9.24).—A catalyst and adsorbent for the preparation of hydrocarbons similar to natural petroleum products consists of a mixture of charcoal and nickel, cobalt, iron, manganese, copper, boron, calcium, sodium, titanium, uranium, or vanadium, and a solid hydrocarbon. A. B. Manning.

Conversion of heavy [mineral] oils into lighter products. M. A. H. de Dampierre (F.P. 590,616, 19.12.24. Conv., 20.12.23).—The heated oil is treated with hydrogen under pressure and the evolved vapours are passed over a large surface of catalyst. A. R. Powell.

Wax sweating and crystallising apparatus. Burmah Oil Co., H. L. Allan, and J. Moore (E.P. 243,447, 29.8.24. Addn. to 208,195; B., 1924, 165).—In an apparatus of the type described in the chief patent, consisting of a number of superposed wax compartments separated from one another by heating and cooling cells, the charging and discharging pipes are located in a space between the sides of the compartments and the container; separate inter-cell charging and discharging pipes are dispensed with. Solid metal fins project downwards from the cells into the wax beneath to increase the heating or cooling effect. T. A. Smith.

Manufacturing lubricating oils. J. W. Weir, Assr. to J. C. Black (U.S.P. 1,564,501, 8.12.25. Appl., 21.3.22).—Lubricating oil stock is treated with sulphuric acid and the greater portion of the sludge removed by settling. Finely comminuted solid absorbent material is then added and the mixture heated until sulphur dioxide is evolved. The absorbent material and absorbed matters are then separated from the stock. T. A. Smith.

Preparation of stable lubricating and insulating oils. F. Frank (G.P. 417,835, 20.2.23).—Refined lubricating and insulating oils before or after use may be improved by treatment with methyl alcohol, which extracts the compounds which are readily decomposed. A. R. Powell.

Dehydration process [for oil emulsions]. H. C. Eddy, Assr. to Petroleum Rectifying Co. of California (U.S.P. 1,565,992, 15.12.25. Appl.,

8.9.24).—An emulsion containing suspended solids is subjected to an electric field to agglomerate and precipitate water and solids, the precipitate is removed, and the residue is filtered to agglomerate the remaining water. H. HOLMES.

Centrifugal dehydrator [for oil emulsions]. C. W. GIRVIN, Assr. to PETROLEUM RECTIFYING CO. OF CALIFORNIA (U.S.P. 1,565,997, 15.12.25. Appl., 3.4.23).—The emulsion is subjected to an electric field to coalesce the particles of water, and the oil is separated centrifugally. H. HOLMES.

Distillation of carbonaceous material. R. M. CATLIN, Assr. to CATLIN SHALE PRODUCTS CO. (Reissue 16,252, 12.1.26, of U.S.P. 1,509,667, 23.9.24. Appl., 18.7.25).—See B., 1924, 933.

Refractory diaphrams for use in surface-combustion apparatus. F. J. COX (E.P. 245,182, 2.9.24).

Combustion apparatus. B. VALJEAN (E.P. 245,497, 6.10.24).

Pulverising and drying materials (E.P. 244,575). —See I.

Combustion of ammonia (G.P. 418,622).—See VII.

Manufacture of hydrogen (E.P. 228,153).—See VII.

Activating decolorising char (U.S.P. 1,565,911). —See XVII.

III.—TAR AND TAR PRODUCTS.

Light oils from low-temperature tars. A. KLEIN (Brennstoff-Chem., 1926, 7, 3—7).—A detailed analysis of the light oils from tar obtained from Saar coals by the Thyssen low-temperature (550°) carbonisation process shows these oils to consist of saturated and unsaturated paraffinoid hydrocarbons with only a very small proportion of aromatic hydrocarbons. B. W. CLARKE.

Reduction of cresols by the Bergius process. F. FISCHER and H. TROPSCH (Brennstoff-Chem., 1926, 7, 2—3).—The reduction of *o*-, *m*-, and *p*-cresol by hydrogen under pressure proceeds very slowly, with the formation of phenol, toluene, and methane and its homologues, showing that a reduction to hydrocarbons as well as the splitting off of the methyl group has taken place. The speed of the reaction is so slow, however, that it is impossible for the phenols formed in the primary decomposition of coal to be appreciably acted upon by hydrogen during the Bergius process. B. W. CLARKE.

Reduction of cresols. G. STADNIKOV, N. GAVRILOV, and A. WINOGRADOV (Brennstoff-Chem., 1926, 7, 7—9; cf. A., 1926, 60).—Cresol passed over finely-divided metallic iron (supported on asbestos) at 500°, is converted almost entirely into toluene. The first effect of the iron is to decompose the cresol, with the deposition of carbon; the carbon thus produced, activated by the iron, then reduces the remaining cresol (92·5% of the whole) to toluene, *p*-cresol being more readily reduced than its isomerides. B. W. CLARKE.

PATENTS.

Tar bitumen emulsions. J. A. MONTGOMERIE (E.P. 244,561, 28.10.24. Addition to 226,032; cf. B., 1925, 91).—A small percentage of soap is added to a bituminous emulsion prepared as described in the chief patent, and a small proportion of melted coal tar or pitch or blast-furnace tar or pitch is incorporated. B. W. CLARKE.

Distilling crude tar oils. G. KRICKHUHN (G.P., 417,974, 25.1.25).—The crude oil mixture from the tar still is led into an oil still, separated from the tar still by a fireproof, heat-insulating material. A. GEAKE.

Separation of the acid fractions, in particular phenols, from mixtures with neutral oils. GELSENKIRCHENER BERGWERKS-A.-G., ABTEIL. SCHALKE, and H. HOCK (G.P. 417,971, 15.8.22).—The oil mixture is treated with a 5—10% solution of sodium carbonate under pressure at about 250°. The phenols dissolve, and the solution of phenoxides is separated from the remaining oils. On cooling the solution the phenols separate, and the carbonate solution can be used for a further extraction. Thus, after a third extraction of a tar oil containing 40% of phenols with 10% sodium carbonate solution at 250°, 83% of the phenols had been removed, only the very feebly acid homologues remaining. A. B. MANNING.

Recovering phenols from ammoniacal liquor (U.S.P. 1,566,795–6).—See II.

Recovering benzol from gases (F.P. 594,531).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Fastness of pigments to light. WAGNER.—See XIII.

PATENTS.

Manufacture of condensation products [dyestuffs] of the anthraquinone series. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 222,125, 16.9.24. Conv., 18.9.23. Addn. to 205,502, B., 1924, 627).—Vat dyestuffs are prepared by condensation of derivatives of benzanthrone substituted in the 1′-position and aromatic acid chlorides by the process described in the chief patent. Yellow dyestuffs are obtained from 1′-methylbenzanthrone or 2-chlorobenzanthrone and benzoyl chloride; the former dyestuff crystallises from nitrobenzene in microscopic spears, m.p. above 300°, and the latter has m.p. 197°. A. J. HALL.

Manufacture of vat dyestuffs from benzanthrone. BADISCHE ANILIN U. SODA-FABR. (F.P. 589,323, 31.10.24. Conv., 10.1.24).—Benzanthrone or 2 : 2′-benzanthronyl is heated with an alkali

alkyloxide or an alcoholic solution of a caustic alkali. For example, a mixture of dibenzanthrone and *iso*dibenzanthrone is obtained by heating benzanthrone at 170° with methyl alcohol and caustic potash. The mixture may be purified by precipitation from sulphuric acid and the constituents separated by taking advantage of the difference in the solubilities of the alkali salts of their leuco-compounds (obtained by reduction with alkaline solutions of sodium hyposulphite); the alkali salt of the leuco-compound of *iso*dibenzanthrone is insoluble. Methyl alcohol may be replaced by *iso*propyl or butyl alcohol. A. J. HALL.

[Manufacture of] vat dyestuffs. KALLE U. Co. A.-G. (F.P. 591,535, 12.1.25).—Vat dyestuffs are prepared by the action of compounds containing reactive hydrogen atoms on chloro-, bromo-, and nitroketoperylene dyestuffs. For example, greenish-blue and reddish-blue dyestuffs are obtained by treating chlorodibenzanthrone and chloro*iso*dibenzanthrone respectively, with phenol in the presence of an alkali. Blue, blue, and greyish-black vat dyestuffs are prepared by heating chlorodibenzanthrone, dichlorodibenzanthrone, and nitrodibenzanthrone, respectively, with *p*-toluidine in the presence of sodium carbonate. A. J HALL.

[Manufacture of] sulphur dyestuffs fast to chlorine. KALLE U. Co. A.-G. (F.P. 588,874, 14.11.24).—Blue to black sulphur dyestuffs fast to chlorine are prepared by heating aromatic dihydroxy-compounds, aminocarbazole, and alkali polysulphides together under pressure. The aromatic dihydroxy-compounds, *e.g.*, quinol, may be replaced by compounds, *e.g.*, benzoquinone, which are converted into dihydroxy-eompounds when treated with polysulphides, and aminocarbazole may be replaced by nitro-, nitroso-, or nitrosonitro-carbazole. A. J. HALL.

[Manufacture of] diphenylmethane dyestuffs. G. L. HUGEL (F.P. 589,745, 9.2.24).—Diphenylmethane dyestuffs are condensed with phenyhmethylpyrazolone, β-naphthol, or nitromethane in aqueous solution and the leuco-products subsequently oxidised. For example, a dyestuff capable of yielding fluorescent shades fast to light on silk and cellulose acetate silk is obtained by condensing Thiopyronine with phenylmethylpyrazolone in an aqueous solution of caustic soda. Under similar conditions golden-yellow (with green fluorescence) and red fluorescent dyestuffs for silk and cellulose acetate silk are obtained from Acridine Orange and phenylmethylpyrazolone and from Thiopyronine and nitromethane respectively. A. J. HALL.

[Manufacture of] greenish-blue dyestuffs [lakes]. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (F.P. 590,464, 13.12.24. Conv., 24.12.23 and 19.5.24).—A greenish-blue lake is obtained by the action of ferric chloride on a solution containing sodium carbonate, sodium acetate, and 8-chloro-1 : 2-naphthylenediamine-5-sulphonic acid (obtained by reduction of the dyestuff obtained by coupling 8-chloro-1-naphthylamine-5-sulphonic acid with diazobenzene chloride) and precipitating the resulting dyestuff by the addition of barium chloride, aluminium sulphate, and barium sulphate. A. J. HALL.

[Manufacture of a] monoazo dyestuff. CHEM. FABR. ROHNER A.-G. PRATTELN (Swiss P. 109,706, 10.5.24).—A bluish-scarlet pigment is produced directly on textile fibres or in substance by coupling diazotised 2-benzamido-*p*-toluidine with 2 : 3-hydroxynaphthoic acid anilide (Naphthol AS). It forms lakes very fast to light. A. J. HALL.

[Manufacture of] dyestuffs from peat. J. KOZAK, M. F. WEINBERGER, AND M. PROKOPCZUK (Austr. P. 99,926, 30.1.24).—Humic acids precipitated by the addition of acids to the product obtained by digesting peat with a dilute solution of sodium carbonate containing caustic soda or potash, are heated at 200° with alkali sulphides or polysulphides in the presence of the salts of heavy metals such as copper, zinc, and iron. The resulting dyestuff may be purified and its colour strengthened by the introduction of nitro-groups. A. J. HALL.

[Naphthalene] chlorination apparatus. S. BROWN, Assr. to HALOWAX CORP. (U.S.P. 1,564,044, 1.12.25. Appl., 13.1.21).—Molten naphthalene is treated with chlorine under reduced pressure to form monochloronaphthalene, and the hydrochloric acid formed is sucked through water and absorbed. Chlorination and absorption proceed satisfactorily under the reduced pressure and there is no risk of leakage from the apparatus. T. S. WHEELER.

Production of anthraquinone derivatives containing nitrogen [isoxazolones]. BADISCHE ANILIN- U. SODA-FABR., Assees. of H. NERESHEIMER (G.P. 418,270, 16.8.23).—Anthraquinoneisoxazolones, which are of use in the manufacture of dyestuffs, are produced by oxidising anthraquinoneisoxazoles not substituted in the isoxazole ring, by treatment in solution or in suspension in acids, with halogens or substances liberating them. For example, chlorine is passed into a suspension of anthraquinoneisoxazole in 62% sulphuric acid at 50—60°, until a filtered test portion dissolves completely in ammonia, yielding a yellowish-green solution. The *product*, of formula, $C_{15}H_7O_4N$, is separated by dilution with water and filtration; it has m.p. 214—215° (crystallised from benzene), and is reduced by alkaline sodium hyposulphite to 1-aminoanthraquinone-2-carboxylic acid. *5-Nitroanthraquinoneisoxazolone* is obtained in a similar manner from the corresponding isoxazole derivative. L. A. COLES.

Preparation of primary aromatic amines. FINOW-G.m.b.H., and H. MÜLLER (G.P. 418,497, 29.3.23).—Aromatic nitro-compounds are reduced to the corresponding amines by treatment with nascent ferrous hydroxide, carbonate, or acetate. For example, an intimate mixture of nitrobenzene with anhydrous sodium carbonate or with flaked sodium hydroxide, is added, with stirring, to ferrous sulphate solution at 30°; stirring is continued for a short time, and aniline is separated by steam distillation. *o*-Nitrobenzaldehyde is reduced

quantitatively to *o*-aminobenzaldehyde by similar treatment at the ordinary temperature. In cases where excess of alkali is not harmful, the reduction can be effected by adding a mixture of the nitro-compound with ferrous sulphate to sodium carbonate solution. L. A. COLES.

Manufacture of azo-dyestuffs. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 245,287, 24.1.25).—See U.S.P. 1,546,328 ; B., 1925, 751.

Condensation products [dyestuffs] of the anthraquinone series. SOC. OF CHEM. IN BASLE, Assees. of P. SCHETELIG (U.S.P. 1,568,627, 5.1.26. Appl., 4.5.25).—See E.P. 234,086 ; B., 1925, 624.

Lakes (F.P. 582,506).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Sulphur content of wool. S. R. TROTMAN and H. S. BELL (J.S.C.I., 1926, **45**, 10—12 T).—Published results for the sulphur content of wool vary from 0·5 to 5%. Whether keratin be either a single protein or a mixture of proteins one would expect it to have a definite and not a variable composition. Variation in results may be due partly to faulty methods of analysis. The authors have examined many published processes, compared with that of Carius, and recommend the following modification of the Benedict–Denis method (J. Biol. Chem., 1909, 363 ; 1910, 401). About 0·2 g. of the wool is digested with pure sodium hydroxide solution made from metallic sodium, till it has just dissolved. A drop of bromine is added and, after a few minutes, the mixture is neutralised with nitric acid, 10 c.c. of the Benedict–Denis reagent are added, and the determination of the sulphuric acid is completed in the usual manner. Different purified wools, of known origin, were analysed by this process. The percentage of sulphur showed but little variation, 3·42% being the maximum, whilst the mean of 40 experiments gave 3·22%. It is contended that the sulphur content of wool is not variable as supposed hitherto, but practically constant.

Proteins of wool. S. R. and E. R. TROTMAN and R. W. SUTTON (J.S.C.I., 1926, **45**, 20—24 T).—The authors have repeated the work of Raikov, Allwörden, and other investigators. They could not obtain any confirmation of Raikov's statement that wool evolves sulphur dioxide when treated with phosphoric acid. Neither Allwörden's "elasticum" nor Justin-Müller's "gelatin" could be isolated. No volatile sulphur compounds were obtained from the solution of wool in sodium hydroxide. When wool is digested with hydrochloric acid, part of it dissolves, leaving a comparatively insoluble residue which dissolves readily in ammonia. From the hydrochloric acid solution and the undissolved residue two different proteins (termed A and B) were obtained, with sulphur contents of 3·67% and 1·49% respectively. After removing protein A from the hydrochloric acid solution, a third was obtained by saturating the filtrate with magnesium sulphate. As the result of experiments made with cold acids and alkalis on wool, it is suggested that protein A is present chiefly in the epithelium, whilst the cortex consists principally of protein B. Protein A is more readily attacked by oxidising agents and chlorine than protein B, and these facts are in accordance with practical experience in the chlorination of wool. On the other hand, protein B being more readliy soluble in alkalis is possibly connected with damage by alkalis.

Chemical analysis of cotton. Waxes of cottons of different origin and their characteristics. L. V. LECOMBER and M. E. PROBERT (J. Text. Inst., 1925, **16**, T 338—344).—The quantities of the carbon tetrachloride extracts (fat and wax) and of the chloroform extracts (fat, wax, and resin), and their analytical characteristics have been determined for a range of cottons. The table summarises the most useful data :—

Cotton	Fat and wax %	Acid value	Saponification value	Unsap. matter %	Iodine value
American (12 samples)	0·435	25	66	64	22
Egyptian (7 samples)	0·39	27	78	54	23
Sea Island (2 samples)	0·51	21·5	61	61·5	23
S. American (5 samples)	0·29–0·54	30	71	60	25
Australian (1 sample)	0·42	29	58	68	19
Mean of these 27 samples	—	26	70	61	23
Indian (6 samples)	0·34	38	121	45	32

Native Indian cottons can obviously be readily distinguished from other growths by the properties of their wax extracts. J. C. WITHERS.

Chemical analysis of cotton. Identification of the fatty ingredients in sized goods. L. V. LECOMBER and M. E. PROBERT (J. Text. Inst., 1925, **16**, T 345—351).—The analytical characteristics of cotton wax (cf. preceding abstract) are sufficiently divergent from those of tallow, Japan wax, spermaceti, castor oil, paraffin wax, and similar "softeners" commonly used in compounding sizes to make it possible to identify these substances in the carbon tetrachloride extracts from sized goods. In the absence of the original unsized cotton, the average values for its particular type may be used for reference. Examples are quoted to prove the utility of the method for cloths containing the above softeners singly or in certain pairs. The chief sources of error are possible adulteration of the softener presumed to have been used, and possible oxidation of unsaturated fats or oils on exposure of the cloth to the air. J. C. WITHERS.

Common faults in cotton goods. F. SUMMERS (J. Text. Inst., 1925, **16**, T 323—337).—The value of microscope technique in elucidating the causes of typical faults is shown. Of chemical interest is a chapter dealing with mercerisation, which shows how

the degree of penetration by the alkali and the efficiency of the tension employed may be judged from cross-sections of the material.

J. C. WITHERS.

Action of ammonia on cotton cellulose. G. BERNARDY (Z. angew. Chem., 1925, 38, 1195—1197).—The action of 22% aqueous ammonia on cellulose does not lead to additive compounds, to adsorption, to entry of an amino- or imino-group into the molecule, to hydrolysis, or to oxidation. It is analogous to, though feebler than, that of other alkalis, and leads simply to the dissolution of alkali-soluble celluloses. When the mixture is heated under pressure, black, amorphous, humin-like substances are also formed, and under prolonged heating at 200° and 40 atm. the cellulose breaks down into a brown powder. The nitrogen content of the cellulose is only very slightly increased by the action of ammonia. The cleansing action of 22% ammonia on raw cotton cellulose is less than that of 1% sodium hydroxide at 100°. Whereas ammonia in this concentration does not cause any swelling of cellulose, permanent swelling is produced by liquid ammonia at −33° to −35°, though unaccompanied by any decomposition. W. T. K. BRAUNHOLTZ.

Lignin. I. Spruce lignin. A. FRIEDRICH and J. DIWALD (Monatsh., 1925, 46, 31—46).—Finely-sieved spruce sawdust was freed from resin by Soxhlet extraction for 7 hrs. with alcohol–benzene (1:1), and from gum by subsequent extraction with cold, aqueous 5% sodium hydroxide for 36 hrs. After removal of the alkali and drying at 60—70°, the wood was triturated with an equal weight of hydrochloric acid (1 vol. of acid, *d* 1·17, to 1 vol. of water). After keeping for 48 hrs. the mixture was boiled for 8—10 hrs. with a tenfold proportion of 96% alcohol, filtered, the filtrate evaporated to one-third of its original volume, and diluted with 10 pts. of water, addition of a little mineral acid improving the filtering properties of the precipitate. This consists of primary lignin, $C_{31}H_{33}O_9(OMe)_5$, and forms, after several purifications by dissolving in alcohol and re-precipitation with water, a pale brown, amorphous, hygroscopic powder, soluble in alcohol, chloroform, acetone, pyridine, carbon disulphide, acetic acid, and ethyl acetate, but insoluble in ether and benzene (cf. Schmidt, A., 1923, i., 274). The yield is 8—10%, calculated on air-dried wood. When dissolved in warm, 2*N*-sodium hydroxide, and re-precipitated with acid after 2 hrs., the primary lignin is partially demethylated with formation of the product $C_{34}H_{35}O_{11}(OMe)_3$, which is soluble in sodium carbonate solution. The methoxyl content of the primary lignin (20·9%) is also reduced (to 16·8%) by treatment with concentrated hydrochloric acid (*d* 1·21), the product being dark brown, insoluble in all media except alkalis, and apparently identical with the lignin described by Willstätter and Zechmeister (A., 1913, i, 955), obtained by the direct extraction of spruce wood with concentrated hydrochloric acid. Treatment of the primary lignin with bromine in acetic acid affords a product containing 4 atoms of bromine, but its formation is accompanied by evolution of hydrobromic acid, and by considerable changes in the constitution of the lignin. Prolonged action of bromine affords successively an alcohol-soluble and an alcohol-insoluble product, each containing 5 bromine atoms, and finally an ether-soluble hexabromo-derivative. Oxidation with dilute alkaline hydrogen peroxide affords a pale yellow, amorphous, compound $C_{28}H_{33}O_9(OMe)_3$, which does not reduce Fehling's solution. Phenylhydrazine converts the primary lignin into a reddish-brown, amorphous product, $C_{30}H_{27}O_7N(OMe)_3$, whilst semicarbazide affords a yellowish brown, amorphous powder, the composition of which is also not in accordance with simple semicarbazone formation. Treatment with excess of methyl sulphate in alkaline solution affords the compound $C_{34}H_{32}O_8(OMe)_6$, yellow-brown, decomp. above 110°, insoluble in alkalis, whilst benzoylation affords the tribenzoate, $C_{55}H_{45}O_{12}(OMe)_5$, pale yellow. This capacity for attaching three benzoyl groups but only one methyl group is possibly to be ascribed to the hydration, in the former case, of an aldehyde group (cf. Powell and Whittaker, A., 1924, i, 375). The typical colour reactions with aniline and phloroglucinol are exhibited by the primary lignin and the methyl derivative. The product of the reaction with aniline corresponds to the addition of 1 mol. of aniline to 1 mol. of the primary lignin, and forms a yellow powder. The colour reaction with phloroglucinol only takes place in presence of hydrochloric acid, and disappears on dilution with water. When the reaction is carried out in alcoholic hydrochloric acid, evaporation of the solution affords a dark reddish-brown product. A bisulphite compound of the primary lignin is described.

F. G. WILLSON.

Lignin. W. KÜSTER and E. SCHNITZLER (Z. physiol. Chem., 1925, 149, 150—172).—Lignin is decomposed by fusion with β-naphthol to give at least two distinct products, one of which, merolignin, is a crystalline chemical individual. The mass is treated with chloroform, filtered, and precipitated by the addition of ether, when a substance is obtained which on drying in a vacuum gives analyses agreeing with the formula $C_{18}H_{13}O_3(OMe)_3$. It is soluble in aqueous potash but not in soda. It melts at 155° (decomp.). The filtrate from the ether precipitation is evaporated, cooled, and treated with light petroleum, when a second precipitate is obtained. This is filtered off quickly, dried, and washed with light petroleum. The dried yellow powder, m.p. 160—168° (decomp.), gives analyses which agree with $(C_{18}H_{14}O_3)_x$. The filtrate is evaporated to dryness, and crude merolignin extracted by digestion of the residue with cold alcohol. Better yields are obtained by extracting the β-naphthol melt direct with petroleum of b.p. 110—130°; the solution is evaporated to dryness and treated with cold alcohol, when crude merolignin is extracted. This may be purified by recrystallisation from chloroform or benzene. It melts at 205—206 Analyses agree with the formula $C_{22}H_{14}O$.

H. D. KAY.

Use of acid hydrolysis in the determination of lignin. L. PALOHEIMO (Biochem. Z., 1925, 165, 463—464).—Replacement of 41% hydrochloric acid by 70% sulphuric acid gives, on hydrolysis, concordant results for the lignin residue, viz., for pine wood 30%, for hay and straw 20%. More accurate investigation shows that these figures are too high. If the time of hydrolysis is too short, carbohydrate material is precipitated along with the lignin, and if too long, humus is formed. By hydrolysing over increasing periods up to one hour, it was shown that the amount of residue was constant for a short time, but then slowly increased, due to adsorption by the lignin of secondary products. This increase could be retarded by use of large amounts of acid. For each 1 g. of material 200 c.c. of acid are used and the residue after half-an-hour is filtered off and submitted to a second hydrolysis for 1 hour. Very difficultly hydrolysable protein material remains, however, after hydrolysis for 24 hrs. A correction for this is applied by subtracting from the lignin figure the protein content as calculated from the total nitrogen of the residue. The accepted figures for pine wood (30%), for clover hay (25%), and for rye straw (20%) are reduced by these methods to 25%, 7%, and 3%, respectively. The amount of lignin is not changed by heating the residue with dilute acid or alkali, nor when such treatment precedes acid hydrolysis. If, therefore, the material receives a preliminary treatment with dilute alkali, which removes most of the substances likely to interfere with the determination, a single acid hydrolysis is sufficient. Lignin of hay is not attacked in the alimentary canal of ruminants. P. W. CLUTTERBUCK.

Formation of sugars as intermediate phase of production of coal from ligneous plant substances. SCHWALBE and SCHEPP.—See II.

PATENTS.

Fireproofing textile fibres and fabrics, and other porous or absorbent substances. T. J. I. CRAIG, and P. SPENCE AND SONS, LTD. (E.P. 244,503, 18.8.24).—The material is impregnated with sodium aluminate and squeezed. It is then immersed for $\frac{1}{4}$—$\frac{1}{2}$ min. in a saturated solution of sodium bicarbonate at a temperature above 50°, but below that at which carbon dioxide is evolved. After squeezing off the excess solution, the material is immersed for $\frac{1}{4}$—$\frac{1}{2}$ min. in a boiling solution of sodium carbonate or other salt, when the alumina is rapidly fixed on the material. The treatment with sodium bicarbonate may be partly or wholly replaced by exposure to gaseous carbon dioxide. A. GEAKE.

Retting flax and similar vegetable fibres. BADISCHE ANILIN- U. SODA-FABR., Assees. of G. PFÜTZER and O. FLIEG (G.P. 419,730, 7.9.24. Addn. to 411,697; B., 1925, 626).—Formamide, or a mixture of formamide and carbamide, is added to the ret water; the time required for retting is thereby reduced. A. GEAKE.

Washing artificial fibres wound upon bobbins. B. BORZYKOWSKI (E.P. 239,482 and 244,324, 9.4.25. Conv., [A] 2.9.24, [B] 13.12.24).—(A) The fibre is wound upon hollow, perforated bobbins, and the washing liquid drawn through it by suction applied to the interior. (B) Coagulating liquor adhering to the fibre is removed by suction before washing as described in (A). After washing, the washing liquid is similarly removed. A. GEAKE.

Manufacture of cellulose derivatives [acetate]. J. O. ZDANOWICH (E.P. 244,148, 15.8.24).—In the production of a solution of cellulose acetate, which can be directly converted into films, filaments, and the like, the acetylation mixture is first chlorinated (cf. E.P. 196,641; B., 1923, 650 A), and a substance containing 20—30% of acetyl obtained. The chlorine remaining in this solution is utilised by adding sulphur dioxide or trioxide, or an oxide of phosphorus. The chlorine compound thus formed acts as a condensing agent for the second stage of acetylation. Acetylation is completed by the addition of a very small quantity (0·01—0·1%) of a powerful condensing agent, such as sulphuric acid. A. GEAKE.

Production of plastic masses. R. GARKE, E. MEYER, and W. CLAASEN (E.P. 241,858, 28.4.25. Conv., 22.10.24).—A mixture of *ar*- and *ac*-tetrahydronaphthyl acetate, or either of these separately, is a solvent for rubber and the like, and also for cellulose esters. These may thus be combined to form solid plastic masses, varnishes, impregnating media for textiles, paper, wood, leather, and the like, and adhesives. A solution of rubber in tetrahydronaphthyl acetate may be added to nitrocellulose before spinning artificial filaments, and the filament then obtained has great resilience and is not affected by water. The addition of a small quantity, *e.g.*, 10%, of tetrahydronaphthyl acetate to rubber hinders atmospheric oxidation and consequent deterioration. The ester is non-volatile at the ordinary temperature and remains in the goods made from it. A. GEAKE.

Manufacture of cellulose esters or ethers in a solvent. N. B. GRILLET, Assr. to SOC. CHIM. USINES DU RHÔNE (U.S.P. 1,566,398, 22.12.25. Appl., 22.11.24).—Cellulose is esterified, in the presence of a liquid solvent of the desired product, in a large, rotating, closed vessel. A. GEAKE.

Prevention of corrosion in steel digesters [for wood pulping]. F. G. RAWLING; dedicated to the citizens of the United States (U.S.P. 1,566,118, 15.12.25. Appl., 21.3.25).—Corrosion of steel digesters for wood pulping is prevented by adding the sodium salt of a weak polybasic acid to the sodium sulphite solution used. Subsequently, during the cooking, aqueous sodium hydroxide is added. A. GEAKE.

Digesting pulp in rotating digesters. E. MORTERUD (U.S.P. 1,566,339, 22.12.25. Appl., 28.5.25).—A tube located in one of the trunnions of the digester is connected to an external heater, and

internally alternately to two tubes leading to opposite ends of the digester. A. Geake.

Manufacture of cellulose. L. Bradley and E. P. McKeefe (Can.P. 246,477, 12.4.24).—Finely divided wood is boiled in a solution of sodium hydroxide, sulphite, and thiosulphate. A. Geake.

Manufacture of paper pulp from vegetable fibres. B. S. Summers (Can. P. 246,537, 16.2.24).—Fibre is boiled in a liquor containing 1—4% of phosphoric acid. A. Geake.

Manufacture of pulp. Maschinenbau-Anstalt Humboldt (G.P. 418,304, 3.6.23).—Fibrous material is first beaten, and while beating is continued solutions of caoutchouc, balata, gutta-percha, or drying, oxidised, or sulphonated oils are added. This procedure avoids the formation of lumps. A. Geake.

Purifying cellulose. C. G. Schwalbe (G.P. 418,976, 19.4.23).—Cellulose is boiled with bases and sulphites in the presence of substances soluble in such solutions, such as carbohydrates, especially sugars, and salts of organic acids. Boiling may be with or without pressure, and preferably with agitation. The above substances may be replaced by molasses or sulphite-cellulose waste liquor. A. Geake.

Treatment of black liquor [from soda-cellulose manufacture]. L. Bradley, Assee. of E. P. Mckeefe (Can. P. 245,831, 1.12.21).—A solution of alkali aluminate is formed in the liquor, the alumina and organic matter are then precipitated together as an intimate mixture, the precipitate is distilled, and the mixture of carbon and alumina obtained is electrolysed in the molten state. A. B. Manning.

Recovering constituents from muds produced in purification of cellulosic alkali lyes with heavy metals. A.-G. f. Anilin-Fabr. (G.P. 419,665, 16.9.24).—Muds containing heavy metals, alkalis, and cellulose are boiled at the ordinary pressure until organic substances are dissolved, and the heavy metal is obtained in a recoverable form. Thus from cellulosic precipitates in viscose manufacture sodium hydroxide is recovered, and also copper as cuprous oxide or metal. A. Geake.

Utilisation of sulphite-cellulose waste liquor. E. L. Rinman (Swed. P. 57,863, 8.6.23).—Sulphite-cellulose waste liquor or spent wash from the distillation of sulphite spirit is evaporated to dryness, and mixed with calcium sulphate and materials containing alumina, ferric oxide, and silica, in such proportion that on ignition sulphur dioxide is evolved and a residue is obtained suitable for the production of a cement. A. B. Manning.

Preparation of alkali cellulose. E. van Weyenbergh, Assr. to Courtaulds, Ltd. (U.S.P. 1,569,692, 12.1.26. Appl., 6.3.25).—See E.P. 237,685; B., 1925, 800.

Grinding stones for grinding wood pulp. Norton Co., Assees. of W. W. Greenwood (E.P. 238,176, 19.12.24. Conv., 6.8.24).

Coal briquettes (F.P. 594,080).—See II.

Improving the odour of methyl sulphide (G.P. 418,129).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Leather dyeing. I. H. Salt (J. Soc. Leather Trades Chem., 1925, **9**, 518—520).—Comparative dyeings on vegetable-tanned leather were made with different acid dyes without added acid or salts, and with addition of sulphuric acid, acetic acid, sodium chloride, sodium sulphate, and sodium sulphate and sulphuric acid respectively. Acid dyes gave as good a colour with acetic acid as with sulphuric acid and in some cases fuller and more even shades. Sodium chloride reduced the colour value of acid dyes on vegetable-tanned leathers. Many acid dyes gave full shades without the addition of acid. Sodium sulphate had a similar action to sodium chloride. Sodium sulphate and sulphuric acid gave slightly paler shades than sulphuric acid alone. D. Woodroffe.

Leather dyeing. II. D. McCandlish and H. Salt (J. Soc. Leather Trades Chem., 1925, **9**, 520—525).—Comparative dye tests on chrome-tanned and vegetable-tanned skivers with a range of acid, basic, and direct dyes have shown that unmordanted chrome leather has a much greater affinity for acid and direct dyes than vegetable-tanned leather, The hydroxyl group in the dyestuff is unimportant. The object of mordanting chrome leather is not to facilitate dyeing but to reduce the attraction between the leather and the dye, thus producing more even shades. A better feel is produced on the finished leather if it has been mordanted. The dyeing of chrome leather with basic dyes is unsatisfactory unless the leather has been mordanted. Dyes with a reddish shade tend to appear more reddish when used on chrome leather. D. Woodroffe.

Patents.

Bleaching agent. Soc. Alsacienne de Prod. Chim. (F.P. 590,876, 22.2.24).—Aromatic sulphonamide derivatives halogenated in the side chain, such as *p*-toluenesulphochloramide, in solution or in admixture with soap, sodium carbonate, or sulphonated castor-oil, are used for cleaning and bleaching vegetable and animal fibres. L. A. Coles.

Preparation of a mordant [for cotton] in place of tannin. L. Sellet (F.P. 589,081, 17.1.24).—Hydrogenated aromatic hydrocarbons are treated with concentrated sulphuric acid, with or without a preliminary oxidation. For example, tetralin is oxidised with bromine water and phosphoric acid, the aqueous layer removed, and the oxidation completed by treatment with air in the presence of ferric chloride as catalyst. Any unchanged tetralin is distilled off and the resinous residue is rendered

soluble in water by treatment with concentrated sulphuric acid. A similar mordant may be prepared by the direct treatment of tetralin with concentrated sulphuric acid, oxidation of the tetralin taking place simultaneously. A. R. POWELL.

Dyeing cellulose acetate products. L. B. HOLLIDAY AND CO., LTD., and A. YOUNG (E.P. 244,936, 24.1.25).—Cellulose acetate products are dyed by the substances obtained by condensing 1-chloro-2 : 4-dinitrobenzene-6-sulphonic acid or 1-chloro-2 : 6-dinitrobenzene-4-sulphonic acid or their salts with substances containing one or more amino- or one or more hydroxy-groups, *e.g.*, aniline. A. GEAKE.

Dyeing cellulose acetate. TEINTURERIE DE LA RIZE (F.P. 590,738, 15.2.24).—Cellulose acetate products are dyed by means of direct cotton dyestuffs after treatment for from 15 min. to 2 hrs. at 50—70° in an aqueous solution containing 1 kg. of barium hydroxide and 1 kg. of barium chloride per 100 litres. A. J. HALL.

Easily soluble preparations of vat dyestuffs. FARBENFABR. VORM. F. BAYER U. CO. (F.P. 589,914, 2.12.24. Conv., 10.12.23, 29.2, and 6.6.24).—Preparations of vat dyestuffs which are soluble in cold or warm water are obtained by mixing unreduced vat dyestuffs with a caustic alkali, a hyposulphite, and a salt of a sulphonic acid capable of acting as a dispersing agent, *e.g.*, sulphonic acids of unsaturated fatty acids, ligninsulphonic acid, sulphite-cellulose waste lye, or water-soluble carbohydrates such as glucose and dextrin; or by the addition of suitable quantities of a hyposulphite and a caustic alkali to an aqueous solution of an alkali salt of a sulphonic acid containing a vat dyestuff. For example, a mixture containing 2-(4′-chloroanilido)-1 : 4-naphthaquinone, Monopole soap (twice sulphonated castor oil), and caustic soda is dried, and sodium hyposulphite added; or the mixture containing the dyestuff, soap, and caustic soda is warmed, filtered, and mixed with caustic soda and sodium hyposulphite; or the dyestuff is mixed with molasses, a caustic alkali, and sodium hyposulphite. A. J. HALL.

Process of dry-dyeing. I. LEVY and L. D. GIMBERG (F.P. 591,075, 16.8.24).—Designs are printed on fabric, paper, and the like by means of a viscous colour paste prepared by adding paraffin wax or a similar substance to a mixture obtained by the addition of an aqueous solution of an organic dyestuff, *e.g.*, Methyl Violet, and lactic acid to a 25—50% solution of a soap derived from stearic acid, the printed fabric being subsequently calendered. Lactic acid may be replaced by citric, tartaric, malic, or oxalic acid. A. J. HALL.

Increasing the fastness to rubbing of dyed materials. CHEM. FABR. A. SCHMITZ (F.P. 592,452, 6.1.25. Conv., 15.9.24).—Dyed animal or vegetable fibres, particularly cotton materials dyed with "ice" colours, are treated with a 1·5—2·0% aqueous solution of a viscous emulsion prepared by treating fats and oils, *e.g.*, castor and olive oils, with sulphuric acid, converting the product into a soap, adding gelatin or glue or similar substance, and mixing the product with a chloro-derivative of a hydrocarbon, such as carbon tetrachloride. The emulsion does not form insoluble soaps when added to hard water. A. J. HALL.

Preparation for finishing natural or artificial fabrics. G. L. BRUGÈRE (F.P. 592,205, 27.3.24).—A mixture of an aromatic amino- or hydroxy-carboxylic acid, formaldehyde, and ammonia is heated in a closed vessel to 90° or 150°. The product is a viscous mass which sets to a jelly when kept in the air. For use in the finishing of fabrics the substance is made into a colloidal suspension in an organic solvent. Treatment with this material increases the strength of the fabric. A. R. POWELL.

Monoazo dyestuff (Swiss P. 109,706).—See IV.

Soaps (F.P. 594,146).—See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Factors governing lime-kiln capacity and fuel economy. V. J. AZBE (Tech. Papers, Nat, Lime Assoc., May, 1925, 37—58).—The regulation of the air supply is the most important factor in determining the efficiency and fuel economy of a lime kiln. A minimum temperature of 900° is necessary to convert limestone into lime, and a small percentage of excess air will be sufficient to lower the combustion temperature below this point. On the other hand, too small a supply of air will produce too high a temperature, giving overburnt lime, and there will be a risk of incomplete combustion. The fuel and air supply must be regulated to give a long, luminous flame, which is much more efficient for transferring heat to the limestone than a short, non-luminous and hotter flame. It is impossible to obtain a regular supply of heat with direct hand-fired kilns, considerable losses occurring at the drawing periods. Properly designed automatic gas producers will give good results; their operation is not improved by steaming, but is improved by passing carbon dioxide from the waste gases. Oil firing with mechanical burners is only economical where cheap oil fuel is available. A determination of the percentage of carbon dioxide and oxygen will be a guide as to the operation of the kiln, using charts developed by the author. The output of lime per cubic foot of kiln space is suggested as a basis for the comparison of kiln capacities. The life of the kiln depends on the type of brick used and the structural design of the kiln. It should be possible to obtain without difficulty 4 tons of lime per ton of fuel burnt, with a thermal efficiency of over 60%. B. W. CLARKE.

Comparative tests of methods for the quantitative determination of sodium sulphide. P. BUDNIKOV (Z. anal. Chem., 1925, 67, 241—248).—A series of analyses of pure and technical grades of sodium sulphide has been made by the following eight methods: gravimetric as barium sulphate,

volumetric after conversion into benzidine sulphate, direct and indirect iodometric titration, titration with permanganate of the amount of ferrous sulphate formed when the sulphide is heated with excess of ferric sulphate, precipitation as cadmium sulphide, direct titration with zinc sulphate, and titration with $0 \cdot 1N$-hydrochloric acid using phenolphthalein as indicator (Podreschetnikov, B., 1908, 21). The last-named method and indirect iodine titration gave the most satisfactory results, and the zinc sulphate method the least satisfactory. The advantage of Podreschetnikov's method is that free sodium hydroxide and sodium hydrogen sulphide may be determined at the same time. The pink colour of the indicator disappears when all the hydroxide and half the normal sulphide has been neutralised; addition of formaldehyde then converts all the sodium hydrogen sulphide into sodium hydroxide, which is titrated as before. From the figures so obtained the composition of the preparation is readily calculated. A. R. POWELL.

Determination of nitrate. K. KÜRSCHNER and K. SCHARRER (Chem.-Ztg., 1925, 49, 1077—1078).—Reduction of nitrates to ammonia by means of finely divided copper and sulphuric acid yields low results owing to imperfect reduction, but accurate results may be obtained if an iron–copper couple is used. 0·5 g. of the nitrate is shaken in a Kjeldahl flask with 3·5 g. of iron wire, 0·5 g. of cupric oxide, and 30 c.c. of 1 : 2 sulphuric acid. After 1 hr., the solution is boiled for 30 min., cooled, treated with sodium hydroxide, and distilled, the ammonia being collected in standard acid. A. R. POWELL.

See also A., Feb., 1926, 112, **X-Ray examination of some ammonia catalysts** (WYCKOFF and CRITTENDEN). 113, **Hydroxides of aluminium and iron** (BÖHM); **Incandescence of metal oxides** (BÖHM). 119, **Adsorption of barium chloride by colloidal hydrated manganese dioxide** (CHLOPIN and BALANDIN). 126, **System water and nitrates and sulphates of ammonium and potassium** (OSAKA and INOUYE); **Separation of manganese and zinc with sodium sulphide** (RUFF and HIRSCH). 127, **Decomposition of metal sulphates by heat** (MARCHAL). 131, **Chemical reactions in powdered mixtures of two kinds of crystals** (TAMMANN). 134, **Catalysis of detonating gas at high temperatures by metals inactive at ordinary temperature** (REMY and GÖNNINGEN). 136, **Decomposition of alkaline-earth phosphates by alkali carbonates** (RAQUET); **Reduction of heavy metal sulphides by barium oxide** (BILTZ and VON MÜHLENDAHL). 138, **Hypochlorous acid and alkali hypochlorites** (VON DIENES). 139, **Preparation of fluorine** (LEBEAU and DAMIENS); **Determination of hydrochloric acid by means of potassium iodate** (KOLTHOFF); **Micro-titration of iodide** (KOLTHOFF); **Acidimetric determination of mercuric chloride** (RUPP and MAISS).

Adsorption of ammonia, sulphur dioxide, and chlorine by wood charcoal. HENGLEIN and GRZENKOVSKI.—See I.

Determination of carbon disulphide. HUFF.—See II.

PATENTS.

Sulphur burner. H. HOWARD, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,566,538, 22.12.25. Appl., 26.12.24).—A receptacle, such as a rotary cylinder, for burning the sulphur is provided with an air inlet and gas outlet and is enclosed in a chamber to which air under pressure is supplied, the air passing partly within and partly without the burner and the combined streams being exhausted through an outlet in the outer chamber adjacent to the outlet in th e burner. B. M. VENABLES.

Manufacture of pure hydrochloric acid. SALZWERK HEILBRONN A.-G., T. LICHTENBERGER, and K. FLOR (G.P. 418,389, 7.2.25).—The acid is produced by treating solutions of alkaline-earth sulphates in fused alkaline-earth chlorides with steam. L. A. COLES.

Manufacture of pure, concentrated nitric acid. BADISCHE ANILIN- U. SODA-FABR., Assees. of C. BECK and O. BALZ (G.P. 418,428, 16.12.24).—Nitric acid containing impurities of higher boiling point is purified by scrubbing it in the form of vapour with boiling concentrated nitric acid. L. A. COLES.

Manufacture of ammonia. C. DEGUIDE (F.P. 591,019, 3.3.24).—Barium cyanide is produced by treating mixtures of barium carbonate, coal, and barium silicate at 1200—1300°, with a counter-current of nitrogen in a rotating tubular retort surrounded by a heating jacket in which carbon monoxide generated during the reaction is burned. The retort is constructed of material of low permeability, such as carborundum, and is maintained under a higher pressure than that prevailing in the outer jacket, to prevent passage of carbon monoxide into the retort. The barium cyanide is decomposed into barium carbonate, ammonia, carbon monoxide, and hydrogen, by treatment at 300—500° with superheated steam in a similar furnace, the carbon monoxide and hydrogen being burnt to supply the necessary heat. L. A. COLES.

Catalysts for the synthesis of ammonia. G. CARRARA (F.P. 591,384. 5.1.25).—Catalytic material for the synthesis of ammonia is worked up into the form of network or is deposited as powder upon network carriers. The network may be rolled up into cylinders. L. A. COLES.

Production of stable bicarbonate of ammonium. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 244,645 21.5.25).—Pure stable ammonium bicarbonate is produced by crystallising it from initially hot solutions and continuously passing a current of carbon dioxide through the solution during the cooling and crystallisation. The salt is separated from the liquor by centrifuging, and dried in air at 80—90°, the mother liquor being used again in the process. The product is improved by adding to the solution, prior to crystallisation, up to about 1% of substances

which influence, and preferably increase, its surface tension; *e.g.*, benzene, petrol, carbon tetrachloride, iron carbonyl, tar oils, ammonium sulphide, or sugar may be added to increase, or naphthalenesulphonic acids substituted in the nucleus by one or more propyl, *iso*propyl, butyl, or *iso*butyl radicals may be added to decrease its surface tension. The product can be stored, *e.g.*, for use as a fertiliser, without any material loss. L. A. COLES.

Combustion of ammonia with oxygen. W. SIEBERT and E. UNGER (G.P. 418,622, 21.10.24).—In apparatus for burning ammonia with oxygen for the generation of heat and light, the gases, in suitable proportions and under suitable pressure, are passed through separate inlets into a mixing chamber which may or may not be provided with nozzles. The mixture is burnt at the mouth of a combustion tube filled or nearly filled with rods or with capillary tubes, which may contain concentric rods constructed of the same or of similar material. L. A. COLES.

Separating zirconium and hafnium halogenides. N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 221,802, 8.9.24. Conv., 15.9.23; cf. E.P. 219,327; B., 1925, 499).—Zirconium and hafnium halides are separated by fractional distillation after the addition of a halide of an element of the fifth or sixth group. For example, 1 pt. of phosphorus pentachloride is added to 2 pts. of a mixture of zirconium and hafnium chlorides. When fused, compounds are formed between the phosphorus and metal chlorides. Between 230° and 360° a fraction rich in hafnium distils over, and above 360° a fraction rich in zirconium. Further separation is effected by repeating the fractionation, and the final products are converted into oxides by treatment with a base. A. GEAKE.

Treatment of [precipitation of tin from] solutions containing alkali oxysalt of tin [and arsenic]. H. HARRIS (E.P. 244,526, 18.9.24).—Solutions containing alkali oxy-salts of tin and arsenic, obtained, *e.g.*, in refining lead, are treated with calcium hydroxide after previous addition of carbonates, *e.g.*, sodium carbonate. Tin compounds are precipitated whilst arsenic remains in solution (cf. E.P. 213,638; B., 1924, 522). L. A. COLES.

Making [lead] arsenates. T. A. MITCHELL and K. TOABE (U.S.P. 1,564,093, 1.12.25. Appl., 21.7.22).—Metallic lead and arsenic trioxide are added to a concentrated solution of arsenic acid containing nitric acid. Lead arsenate is precipitated, the concentration of the arsenic acid remaining constant. At intervals the precipitate is removed and the arsenic acid solution concentrated and used again. T. S. WHEELER.

Production of aluminium chloride. L. BURGESS, Assr. to M. BARNETT (U.S.P. 1,566,269, 22.12.25. Appl., 27.7.21).—Aluminium chloride is produced by treating material containing aluminium silicide with hydrogen chloride. L. A. COLES.

Manufacture of nickel salts. W. N. KOHLINS (Can. P. 244,129, 6.6.23).—Solutions of commercial nickel in sulphuric acid are treated with oxidising agents to convert ferrous salts into ferric salts, and, after addition of nickel carbonate, are filtered and concentrated to crystallisation. L. A. COLES.

Neutralising titanic acid obtained by hydrolytic precipitation from titanic sulphate. FABR. DE PROD. CHIM. DE THANN ET DE MULHOUSE (F.P. 590,743, 15.2.24).—Freshly precipitated titanic acid is treated with aluminium hydroxide or zinc hydroxide, or with sodium aluminate or zincate, whereby any sulphuric acid present is converted into soluble sulphates, which are removed by washing. Small quantities of aluminium hydroxide or zinc hydroxide are retained by the titanic acid, and neutralise residual traces of acid not removed during the washing. L. A. COLES.

Manufacture of barium sulphide. C. DEGUIDE (F.P. 592,805, 4.4.24).—Barium sulphide is produced by heating barium sulphate in the presence of a reducing gas, the gas being burnt to provide the necessary heat, without the flame coming in contact with the barium sulphate. L. A. COLES.

Rendering common salt suitable for table and industrial use. M. KRÜGER and S. R. UNKEL (F.P. 593,279, 12.8.24. Conv., 2.8.24).—Crude sodium chloride is purified by repeated washing with a counter-current of water or more or less saturated mother-liquor. L. A. COLES.

Manufacture of metal [*e.g.*, copper] sulphate solutions. F. KLEINMANN (G.P. 418,723, 24.6.23).—Copper and other metal sulphates are produced by treating the finely-divided metal in a tower-shaped apparatus, with a stream of sulphuric acid containing oxides of nitrogen, the reaction products being continuously oxidised by a counter-current of air and steam, which is injected into the apparatus at such a temperature that the reaction velocity increases, or at least does not diminish, until the sulphate solution flows out of the apparatus. After separation of the sulphate crystals, the mother-liquor is mixed with fresh sulphuric acid and used again. The gas outlet of the apparatus can be throttled so as to produce sufficient pressure to force the copper sulphate solution through a filter in the lower part of the apparatus. L. A. COLES.

Treatment [purification] of zinc sulphate solutions. AKTIEBOLAGET OSKARSHAMNS KOPPARVERK (Swed. P. 57,951, 19.1.22).—Zinc sulphate solutions for use in the production of electrolytic zinc are treated with precipitated copper, copper sulphate, and a reducing agent or cuprous oxide, whereby chlorides are precipitated as cuprous chloride. L. A. COLES.

Apparatus for effecting recovery of heat and water vapour in the catalytic manufacture of hydrogen by the action of water vapour upon carbon monoxide. G. L. E. PATART (E.P. 228,153,

16.1.25. Conv., 21.1.24).—Carbon monoxide or gas containing it is preheated and charged with water vapour by passage upwards through the lower of two sections of a tower, in which it is scrubbed by a descending stream of hot water, and on leaving the section at the top, steam is injected into the gas to increase its moisture content, and the mixture is passed into a catalyst chamber for the production of hydrogen. On leaving the chamber, the gases pass upwards through the upper section of the tower, in which they are cooled and deprived of the greater part of their moisture content by scrubbing with cold water. Water is circulated continuously through both sections of the tower, which are provided with bubbling plates or other means for effecting intimate contact between the gas and the liquid, the hot water collecting at the bottom of the upper section, passing through a siphon tube to a spraying device at the top of the lower section, and the cold water collecting in a constant-level reservoir below the lower section being pumped to the top of the upper section. L. A. COLES.

Production of pure nitrogen from combustion gases. N. CARO and A. R. FRANK (G.P. 418,495, 14.2.23).—Catalysts for use in the production of pure nitrogen from combustion gases contain copper, cobalt, chromium, iron, uranium, and other heavy and noble metals, and their oxides, in varying proportions, deposited upon non-reacting porous carriers. Carbon dioxide is subsequently removed from the nitrogen by absorption. L. A. COLES.

Apparatus for production of hypochlorites and chlorates. M. WILDERMAN (U.S.P. 1,568,787, 5.1.26. Appl., 13.4.21).—See E.P. 183,671 ; B., 1922, 812 A.

Treating sulphide ores and concentrates (U.S.P. 1,566,379).—See X.

Electrolysis of alkali chlorides (U.S.P. 1,565,943).—See XI.

VIII.—GLASS; CERAMICS.

Analysis of recent measurements of the viscosity of glasses. II. G. S. FULCHER (J. Amer. Ceram. Soc., 1925, 8, 789—794).—The method of analysis described in a previous paper in relation to three-component glasses (cf. B., 1925, 590) has been applied to two series of four-component glasses, namely, soda-lime-magnesia-silicate glasses and soda-lime-alumina-silicate glasses. The first of these was formed by the molecular substitution of MgO for CaO in the glass $6SiO_2$, 1·15 Na_2O, 0·84 CaO, and the second by substituting Al_2O_3 for CaO in the glass $6SiO_2$, $1·11Na_2O$, 0·95CaO. In the first series, the aggregation temperature, T_a, falls to a sharp minimum when the ratio MgO : CaO is 1·13, corresponding to the composition $6SiO_2$, $1·15Na_2O$, 0·445MgO, 0·395 CaO. In the second series, a similar sharp minimum is observed for the composition $6SiO_2$, $1·11Na_2O$, 0·81CaO, $0·14Al_2O_3$. F. SALT.

Calcining as an aid to grinding [flint]. W. M. MYERS (J. Amer. Ceram. Soc., 1925, 8, 829—842).—Comparative grinding tests were conducted on calcined and on uncalcined flint, care being taken to ensure uniformity in the grinding conditions ; *e.g.*, all material from the jaw crusher was passed through a meshed feed to the pebble mill. A similar series of tests was carried out on calcined flint, part of which had been quenched in cold water, and the remainder allowed to cool slowly in air. The advantage of calcining was most marked in material ground for 6 hrs. in the pebble mill. The improvement however, scarcely warrants the additional cost of calcining. The use of more suitable crushing equipment will, in the future, probably tend to eliminate the calcining operation. F. SALT.

Constitutional changes occurring in clays on heating. RESEARCH STAFF OF GEN. ELECTRIC CO. (J. F. HYSLOP) (Trans. Ceram. Soc., 1924–5, 24, 402—406).—The *X*-ray diffraction patterns of various clays, *e.g.*, china clay, silicious clays, ball clay, show that mullite, not sillimanite, is formed at temperatures above 1050°, as well as transformed quartz or cristobalite. The temperature at which complete stability is reached varies with the nature of the clay. Kaolinite breaks down at 600° to form a silicate, presumably Al_2O_3,SiO_2, stable to 900° ; above this mullite is formed. B. W. CLARKE.

Thermal expansion of refractories. F. H. NORTON (J. Amer. Ceram. Soc., 1925, 8, 799—815).—A number of materials were tested for thermal expansion or contraction up to 1700°. The tests were conducted on specimens 9 in. long and 1 in. square in a gas-fired furnace with a neutral atmosphere. The length of the test-pieces in the furnace was measured directly with filar telescopes. The materials tested included silica, kaolin, fireclay, silicon carbide, zircon, zirconia, mullite, magnesite, chrome, spinel, lime (calcined between 1850° and 1900°), fused alumina, and insulating bricks. In a series of tables, these materials are arranged, in order of merit, in accordance with their m.p., mean coefficient of expansion, maximum coefficient of expansion between 300° and 700° (regarded as giving an indication of resistance to spalling), and temperature at which irreversible contraction begins. F. SALT.

Fractional fusion of refractories. R. HUSTIN (Chim. et Ind., 1925, 14, 691—692).—Bricks which had done service in a reheating furnace, expecially those in the hearth, were found, on examination after 8 days, to have undergone partial fusion at the exposed surface. The fused, surface portion proved, on analysis, to be much richer in alumina and fluxes than the unaltered portion of the brick. This change is shown to be due to a reaction between the fluxes introduced by the coal and the free silica in the brick, resulting in the formation of fusible silicates. A protective coating, rich in alumina, is thus formed on the surface. This is gradually destroyed and a new surface becomes exposed, whereupon the process

is repeated. The fusion process is thus assumed to take place in intermittent stages and not continuously.
F. Salt.

Use of silica refractories. P. B. Robinson (J.S.C.I., 1926, **45**, 29—33T).—The constitution and thermal properties of silica bricks of different specific gravities are described, and illustrated by photomicrographs and diagrams correlated with expansion curves, and methods of overcoming expansion troubles are outlined. Silica brick has a higher thermal conductivity and greater refractoriness under load than fireclay ; this allows of a greater throughput in carbonisation plants. By suitable selection and treatment of bricks, spalling troubles and the abrasion of the brick due to hot dusty gases can be reduced. Bricks should be stored in a dry place. Jointing material must be of refractoriness comparable with that of the brick and give sound adhesive joints at all temperatures.

Laboratory load furnace [for testing refractories]. P. D. Helser (J. Amer. Ceram. Soc., 1925, **8**, 822—825).—An Ajax-Northrup type of high-frequency induction furnace is so constructed that a load can be applied directly to the test-piece, which is held between two anvils and supported on a scale platform. The resistance material may be either graphite or molybdenum. Change of volume in the specimen can be followed throughout the test by means of an Ames dial. F. Salt.

Porcelain for high-tension insulators. K. H. Reichau (Trans. Ceram. Soc., 1924-5, **24**, 279—301).—Porcelain for high-tension insulators must be a dense, homogeneous body, with high electrical resistance, and considerable mechanical strength. It must be able to withstand rapid temperature changes and be stable under varying atmospheric conditions. Correct proportioning and thorough mixing of the quartz, china clay, and felspar are essential to produce a homogeneous porcelain which will not suffer distortion on firing; the size of the silica grains is most important. A fired cylindrical test-piece, about 6—8 in. in diameter and the same height, should show a dense structure on fracture, and a thin section should show a felted network of sillimanite crystals with very little free silica. The extent of the formation of sillimanite can be estimated by the degree of resistance to hydrofluoric acid. The electrical and mechanical tests of the porcelain must be varied according to the work the insulator is to do ; in many cases dynamic tests are more useful than static tests. B. W. Clarke.

Metal porosimeter for determining the pore volume of highly vitrified ware. L. Navias (J. Amer. Ceram. Soc., 1925, **8**, 816—821).—A porosimeter of the McLeod gauge type is constructed of steel to meet the requirements of routine work, for which the glass apparatus is too delicate. The receptacle is in two parts, the lower being made of ordinary steel, and the upper, or cap, of 20% chrome steel, to which the glass capillary can be welded. The results of porosity determinations vary considerably according to the fluid medium used, whether air, water, etc. The term "pore volume" is suggested to distinguish the results of the air method from those obtained by the absorption method. F. Salt.

Method for measuring porosity [of ceramic materials etc.]. P. B. Robinson (J.S.C.I., 1926, **45**, 33—34T).—The material is coated with an impervious layer of plasticene, and after weighing in air and in water the bulk density is calculated. From this and the powder specific gravity of the material, the porosity can be calculated.

Testing the cross-bending strength of enamels. R. R. Danielson and W. C. Lindemann (J. Amer. Ceram. Soc., 1925, **8**, 795—798).—An apparatus for measuring the bending strength of enamelled steel sheets consists of stationary supports and a movable roller fulcrum, which is raised vertically in applying the load, by an eccentric arrangement operated by a ratchet wheel. F. Salt.

Laboratory high-temperature kiln. Norton. —See I.

Refractories for gas retorts. Emery.—See II.

Sintered glass crucibles. Elsdon.—See XVII.

Patents.

Utilisation of domestic and industrial refuse [for making glass]. A. Grote (E.P. 244,358, 16.7.25).—The silicious material separated from town refuse by screening is sintered and is charged into a smelting furnace together with the clinker obtained by the combustion of the coarse screenings. The charge is treated with a mixture in suitable proportions of substances containing silica and saline constituents, *e.g.*, quartz, sand, firebrick, potash, soda, Glauber's salt, in quantity such that a product suitable for glass-making is obtained directly by fusion. A softening agent, such as red lead, and colouring agents, such as cobalt, cupric oxide, iron, uranium oxide, chromium oxide, cuprous oxide, or bone meal, may be added to the charge.
H. Holmes.

Manufacture of abrasives. C. Gergonne (G.P. 417,888, 4.10.24).—Slags containing alumina, *e.g.*, from aluminothermic or thermoelectric processes, are fused, preferably in an arc furnace, so that the size of the crystal grains is increased. Reducing agents are added during the fusion process, so that on cooling and grinding, reduced iron can be removed magnetically, and other impurities by screening, hand sorting, etc. B. W. Clarke.

Manufacture of sheet glass. E. C. R. Marks. From E. Danner (E.P. 245,362, 30.6.25).

Cleaning chalk-flint stones. W. Noake (E.P. 245,613, 30.1.25).

IX.—BUILDING MATERIALS.

Wood preservation. C. Schantz (Brennstoff-Chem., 1926, **7**, 1—2).—The preservative effect of creosote on timber is due to the poisonous nature of

phenol and the cresols, whereby insects and fungoid growths are destroyed, and to the water-repelling property of the tar oils, whereby atmospheric attack is resisted and decay prevented. In general, it is only the sap wood which is affected, and in order to obtain the maximum effect with the minimum consumption of creosote, it is only necessary to impregnate completely the cells of the sap wood. It is shown how this optimum impregnation may be calculated for pine wood from the specific gravity of the wood itself and of the actual woody tissues; further the life of the preserved timber only increases asymptotically after a certain degree of impregnation, and consideration of this fact enables considerable economy in creosote to be effected.

B. W. Clarke.

Determination of the quality of a concrete. R. Stumper (Bull. Soc. chim. Belg., 1925, 34, 296—303).—A method is given for calculating the sand : cement ratio in a concrete from determinations of the silica, lime, and loss on ignition of the concrete, cement, and sand respectively. A simpler method requires the silica or lime values only of the three components, the solution being partly graphic. The first method is shown experimentally to be the more exact.

A. Cousen.

Factors governing lime-kiln capacity and fuel economy. Azbe.—See VII.

Patents.

Manufacture of fused cement. Soc. des Ciments Français, and Bureau d'Organisation Economique, Assees. of J. Bied (E.P. 225,858, 4.12.24. Conv., 4.12.23).—The raw materials are subjected to dehydration and decarbonation, at a temperature insufficient to cause partial fusion, and then conveyed to a separate fusion furnace. Partial fusion and blockage in the conduit pipe is prevented by providing a separate passage through which the hot products of combustion (at 1400—1500°) used to heat the fusion furnace are conveyed.

B. W. Clarke.

Making cement and mortar. O. Simon (E.P. 244,603, 22.1.25).—Fine meal (ground to pass a mesh of 10,000 per sq. cm.) of stone, cement, slate, slag, etc. is added to cement for waterproofing purposes. In addition, oils, fats, albumins, bitumen, etc. may be used for this purpose; the rate of setting of the product may be regulated by the addition of gypsum meal or an alkali lye, or chlorides of alkali or alkaline-earth metals. Bituminous mixtures are improved by the addition of acid. The density of the product is increased by passing an electric current through the mass while setting.

B. W. Clarke.

Concrete material. K. P. Billner (E.P. 244,671, 22.7.25).—Cement to which powdered zinc or aluminium has been added in the proportion of 0·03—0·2%, is mixed to a grout with water and lime, with or without the addition of fine aggregate. Screened coarse aggregate is added to the grout mixture, forming a porous or expanding concrete material owing to the bubbles of gas evolved from the metal.

B. W. Clarke.

Manufacturing cement. C. Leonardt (U.S.P. 1,563,755, 1.12.25. Appl., 19.11.23).—Limestone is treated with hydrochloric acid, asphalt or heavy oil is added, and the mixture incorporated with cement, which it renders waterproof and plastic.

T. S. Wheeler.

Tar bitumen emulsions (E.P. 244,561).—See III.

Utilising sulphite-cellulose waste liquor (Swed. P. 57,863).—See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Thermal treatment of electrolytic iron in a vacuum and recovery of gases. R. Hugues (Rev. Mét., 1925, 22, 764—775).—A volume of gas equal to two and a half times the volume of iron is obtained by heating electrolytic iron *in vacuo* for 2 hrs. at 1500°. At 730° the volume of gas liberated is one and a half times the volume of metal. The gas contains CO_2 6%, CO 34%, H_2 49—51%, hydrocarbons 4—2%, other gases 7%, and O_2 nil. The curves showing evolution of gas as a function of the temperature indicate the break up of a definite quantity of hydrogen–iron complex for each temperature. The magnetic properties appear to be directly related to the liberation of gas, the point of minimum remanent magnetism corresponding to the maximum quantity of gas evolved. The evolution of gas causes at 100° a progressive variation in the mechanical properties which is not marked by a change in the microstructure. This variation commences before any crystalline change and yields a curve similar to that for evolution of gas as a function of the temperature for the same period of heating. A change in the crystal structure commences at 620° and also a further variation in the mechanical properties. There is a sharp variation in the curve between 730° and 910° due to the combined effects of structural change and evolution of gas on the mechanical properties. The hydrogen–iron complex, which is hard and brittle, is believed to be a compound in solution in iron.

M. Cook.

Analysis of precious metal doublé, solder-filled wire, etc. A. Sauerland (Chem.-Ztg., 1925, 49, 1078—1080).—As the gold layer in gold–silver or gold–base metal doublé rarely contains less than 40% Au and is therefore not attacked by nitric acid, the silver or base metal layer may be removed from 1 g. of the doublé by treatment with 25 c.c. of 1 : 4 nitric acid, with the addition of tartaric acid if the base metal contains tin or antimony. The gold layer is washed, dried at 120—150°, weighed, and assayed for gold and silver by cupellation. The nitric acid solution is analysed as usual for base metals and silver. Doublé of standard silver and base metal alloy (brass, tombak, or nickel-silver) may be separated by anodic solution of the base

metal layer in 1 : 5 sulphuric acid, using a platinum cathode, or by treating the doublé with very dilute *aqua regia*. The silver coating of solder-filled silver wire may be removed by dipping the wire for a few seconds in a mixture of 5 c.c. of strong sulphuric acid and 0·5 c.c. of strong nitric acid. A second layer of nickel-silver may be removed by means of dilute *aqua regia*, leaving a thin core of solder which is analysed as usual. The gold layer of platinum-gold doublé can be removed only by anodic solution in 5% potassium cyanide solution, in which platinum is insoluble, but the method is very slow. It is preferable therefore to dissolve the whole sample in *aqua regia* and determine the gold, silver, platinum, and palladium. The silver chloride is reduced by heating in a current of coal gas, and after weighing, the silver is dissolved in nitric acid, any platinum residue being dissolved in *aqua regia* and added to the main solution. Gold is removed with sodium hydrogen sulphite, the filtrate is oxidised with chlorine, the platinum and palladium are precipitated together by addition of ammonium chloride and alcohol, and the copper is determined by precipitation with thiosulphate. The sum of the platinum and palladium gives the weight of the platinum layer; that of the gold layer is found by difference. If the gold layer consists of "white gold," palladium and platinum may form part of this alloy, in which case the palladium and platinum in the gold layer are separately determined in some thin filings scraped from the lower surface of the doublé and allowance is made for the amount so found in calculating the weights of the two layers. A. R. POWELL.

Separation of the platinum metals. L. WÖHLER and L. METZ (Z. anorg. Chem., 1925, **149**, 297—323).—By distillation with acid permanganate or with chromic and sulphuric acids, osmium is separated from all metals, except ruthenium, as the volatile tetroxide. Tellurium remains in the residue as the non-volatile telluric acid. Separation from ruthenium is effected by heating at 275° in a current of nitrous gases. Oxidation to the volatile osmium tetroxide takes place more readily than in oxygen, because in the latter, gas oxidation is hindered by the formation of the non-volatile dioxide. Tellurium is not volatile below 500°, or ruthenium below 600°. Volatilisation of osmium is hindered by sulphur and sulphides, and a higher temperature is then requisite; separation is still quantitative. Osmium is determined in the nitrous distillate by reducing with Devarda's alloy and distilling with permanganate or chromic acid. In nitrous gases tellurium forms the monoxide as an unstable intermediate product. When heated in nitric oxide to 520°, osmium forms the dioxide, OsO_2. Rhodium and iridium are separated by chloridising with excess of sodium chloride at 650°, when the double salts Na_2IrCl_6 and Na_3RhCl_6 are obtained. These are separated by extracting with a mixture of equal volumes of acetone and ether, in which the iridium salt is soluble. Rhodium is also separated from iridium and ruthenium by fusing with more than 20 times its weight of bismuth. A rhodium-bismuth alloy is formed which is completely dissolved by hot 50% nitric acid. When less bismuth is used a new crystalline *compound* $RhBi_2$, is formed. Ruthenium is separated from iridium and rhodium by fusing with sodium hydroxide at 550°. The greater part of the ruthenium is converted into sodium ruthenate, and the remainder into ruthenium dioxide, which is soluble in nitric acid. Iridium forms the dioxide and rhodium the sesquioxide, both of which are insoluble in nitric acid. Iridium is partially dissolved by molten potassium hydroxide and, to a greater extent, by potassium carbonate, on account of its higher m.p. Ruthenium is also separated from rhodium and iridium as the volatile tetroxide by distillation with sodium hydroxide solution and chlorine. If it is alloyed with iridium it is necessary first to convert it into the soluble ruthenate by repeated fusion, preferably with potassium hydroxide and potassium nitrate. The platinum metals can therefore be separated as follows. Osmium is volatilised in nitrous gases, and platinum and palladium extracted from the residue with *aqua regia*. From the solution platinum is precipitated with ammonium chloride. Rhodium is removed from the undissolved residue by fusion with bismuth, and the iridium and ruthenium are separated by fusion with sodium hydroxide or by distillation with alkali chloride. The order of removal of rhodium and ruthenium may be reversed.

A. GEAKE.

System aluminium–zinc. O. TIEDEMANN (Z. Metallk., 1926, **18**, 18—21).—After ageing for 6 weeks at the ordinary temperature alloys of zinc and aluminium containing 0—20% Zn, which have been annealed at temperatures between 250 and 500° for several hours, characteristic changes take place in the ductility and tensile strength so that the field of the aluminium–zinc solid solution may be divided into four parts according to the behaviour of the alloys during the above treatment. Field I. is bounded by curved lines joining the points (560°—0% Zn) and (275°—6% Zn) and the latter point with (0°—3% Zn), field II. by the first line, the horizontal at 275°, and a second curved line joining the points (560°—0% Zn) and (275°—17% Zn), field III. by the last-named line, the solidus, and the boundary between the α solid solution and the heterogeneous region, and field IV. by the continuation of this boundary below 275°, the 275° horizontal, and the lower curved line of field I. Alloys containing less than 3% Zn in field I. consist entirely of β solid solution, which remains unchanged on ageing, whilst only a small increase takes place in the hardness and ductility. Alloys containing 3—6% Zn are in this field above 250° and in field III. below this temperature, so that, on ageing them after quenching, the β solution slowly breaks down into a "new crystal form" and the tensile strength is increased whilst the ductility shows a slight increase (quenched from above 350°) or decrease (quenched from below 350°). Alloys in field II. contain α and β solid solutions in equilibrium but, on cooling below 275°, field IV. is entered and the alloys then consist of β solid solution and the "new crystal form";

consequently alloys quenched from within this field and then aged show a marked increase in tensile strength but a great decrease in ductility. Alloys quenched from field III. (α solid solution) show similar alterations in tensile strength and ductility on ageing to those in field I. containing 3—6% Zn. On ageing alloys containing more than 8% Zn quenched from temperatures below 275° in field IV. a considerable increase in the ductility and a marked decrease in the tensile strength take place owing to decomposition into β solid solution and free zinc. A. R. Powell.

Annealing, quenching, and reheating of some industrial nickel-brasses. F. M. Ostroga (Rev. Mét., 1925, **22**, 776—786).—The three alloys investigated, containing Cu 50·42%, Ni 6·25%; Cu 44·70%, Ni 10·34%; and Cu 39·65%, Ni 14·75%, consist at ordinary temperatures of α and β. Differential dilatometric curves (Chévenard) do not yield any evidence of an abrupt change below the α-β inversion point, but indicate a progressive transformation similar to that found in ordinary brasses. On cooling, two points are indicated, the position of the higher one being little affected by the cooling rate, while the lower one is depressed to 100° by cooling in air. The martensitic structure has not been obtained in quenched alloys. Heating to 200° has no appreciable effect on the hardness; heating to 200—300° causes a slight diminution; the value remains constant in alloys heated to 300—550°, and heating above 550° results in a rapid fall in hardness. Quenching in oil or water from high temperatures renders the alloys excessively brittle. Maintaining the alloys at high temperatures is distinctly harmful, since the crystals grow rapidly and produce a coarse structure and brittleness. The alloys are correctly hardened by water-quenching from 600° or oil-quenching from 775° and reheating to 550°. The reheating to 550° results in a greater hardness than oil-quenching from 775° without further treatment. M. Cook.

Copper containing bismuth. W. Stahl (Metall u. Erz, 1925, **22**, 421—422; Chem. Zentr., 1925, II., 2324—2325).—Copper containing 0·02% Bi as metal is red-short whereas that containing 0·05% is appreciably cold-short. If the bismuth is converted into antimonate or arsenate 0·06% Bi does not render the metal red-short. In order to overcome the deleterious effect of small amounts of bismuth in copper, the metal is melted with the requisite amount of copper–arsenic alloy, allowed to oxidise, and finally poled until the oxygen content is reduced to 0·08%. After this treatment the metal has all the mechanical properties of tough-pitch copper.
A. R. Powell.

Viscosity of alloys at high temperature. J. Cournot and K. Sasagawa (Rev. Mét., 1925, **22**, 753—763; cf. B., 1925, 994).—An apparatus is described for determining the rate of flow and the viscosity limit of metals, and details are given of a special compensating device to eliminate errors due to the diminution of cross-section of the test-piece with elongation. In the work of previous investigators this source of error has not been taken into consideration. The test-pieces were 1 sq. mm. in section and 100 mm. long and were heated in an electric furnace in an atmosphere of nitrogen. The amount of elongation produced by a given load at definite temperatures has been plotted against time for several carbon and alloy steels and the viscosity limits are deduced from these curves. The time of application of the load was 1½ hr. For a soft carbon steel the limit of viscosity is 19·7 tons/in.² at 350° and falls to 2·5 tons/in.² at 600°. The limit of a nickel–chromium alloy falls from 26·7 tons/in.² at 550° to 1·9 at 850° and that of silicon–chrome steel from 38·1 at 500° to 0·6 at 800°. The increase in the rate of flow due to small additional load is more pronounced as the temperature is raised and for special steels at 800° is less than it is for ordinary steels at 600°. The importance of the viscosity limit in fixing factors of safety is emphasised. M. Cook.

Recrystallisation [of metals]. H. Hanemann (Z. Metallk., 1926, **18**, 16—17).—A further discussion of the results of earlier work (cf. B., 1925, 674, 925), in which it is shown that the harder a metal is at the temperature of rolling the greater is the amount of work required to obtain a predetermined grain size after annealing. If, however, the metal is subjected to too severe a rolling the grain size after recrystallisation will not be appreciably reduced and the desired improvement in the mechanical properties of the metal will not be produced. On the other hand, too gentle rolling may result in a coarse grain structure owing to the amount of work expended being below the minimum necessary to produce recrystallisation. The latent energy induced in the metal by rolling is proportional to the degree of rolling and therefore inversely proportional to the grain size after recrystallisation. The fact that recrystallisation of a worked metal commences along the slip planes and that the cause of hardening is also to be found in the slip planes indicates that there is a definite relation between the grain size of a metal after recrystallisation and the hardness induced by deformation.
A. R. Powell.

Present position of the electric furnace for the melting of non-ferrous metals. M. Tama (Z. Metallk., 1926, **18**, 7—14).—A review of the present position of electric furnaces, especially induction furnaces of the Ajax–Wyatt and Northrup type, for use in the melting of non-ferrous metals and alloys, in which are briefly discussed the effects of the electrical resistance, volatilisation point, specific heat, and viscosity of brass on the design of the furnace and its efficiency and on the life of the refractories. A. R. Powell.

Tensile properties of single iron crystals and influence of crystal size on the tensile properties of iron. C. A. Edwards and L. B. Pfeil (J. Iron and Steel Inst., 1925, **112**, 79—110).—See B., 1925, 806.

Orientation of crystals produced by heating strained iron. C. F. ELAM (J. Iron and Steel Inst., 1925, **112**, 111—112).—See B., 1925, 806.

Dilatation of cast iron during repeated heating and cooling. J. H. ANDREW and R. HIGGINS (J. Iron and Steel Inst., 1925, **112**, 167—189).—See B., 1925, 806.

Magnetic and electrical properties of cast iron. J. H. PARTRIDGE (J. Iron and Steel Inst., 1925, **112**, 191—224).—See B., 1925, 805.

Direct process for manufacture of steel. H. FLODIN (J. Iron and Steel Inst., 1925, **112**, 9—25). —See B., 1925, 807.

Effect of temperature on the behaviour of iron and steel in the notched-bar impact test. R. H. GREAVES and J. A. JONES (J. Iron and Steel Inst., 1925, **112**, 123—165).—See B., 1925, 807.

See also A., 1926, 111, **X-Ray analysis** (GÜNTHER and STRAUSKE). 112, **X-Ray examination of α-iron plastically strained** (ONO); **X-Ray examination of mechanism of crystal rearrangement and cause of strain hardening** (ONO); **Allotropy of chromium** (BRADLEY and OLLARD); **X-Ray examination of gold–copper and palladium–copper mixed crystals** (JOHANSSON and LINDE); **Density of tungsten** (DAVEY); **Arrangement of micro-crystals in rolled platinum plate** (TANAKA). 117, **Allotropy of zinc** (STOCKDALE); **Vapour pressures of metals** (RODEBUSH and DIXON). 119, **Alloys of titanium and aluminium** MANCHOT and LEBER). 131, **Catalytic phenomena when zinc is dissolved in acids** (CENTNERSZWER and STRAUMANIS). 133, **Passivity, catalytic action, etc.** (RUSSELL). 135, **Electrolytic deposition of nickel** (SAXON). 138, **Solubility of ruthenium in hypochlorite solutions** (HOWE and MERCER); **Behaviour of carbon monoxide towards salts of palladium and platinum** (MANCHOT). 140, **Determination of copper** (AZZALIN); **Differential reaction of cerium** (FERNANDES); **Electrometric determination of iron with bromate** (COLLENBERG and SANDVED). 141, **Benzoylmethylglyoxime as precipitant of palladous salts** (HANUS, JILEK, and LUKAS); **Separation of iridium from rhodium and platinum, and separation of platinum and rhodium** (WADA and NAKAZONO).

Fractional fusion of refractories. HUSTIN.—See VIII.

Rust-protective power of paints. HERRMANN. —See XIII.

PATENTS.

Treating [iron] ores. W. R. VAN SLYKE (U.S.P. 1,565,689, 15.12.25. Appl., 7.3.21).—The ore is sintered to obtain a porous clinker which is then smelted with a non-porous fuel.

A. R. POWELL.

Purification of cast iron. J. G. PLATON (F.P. 591,053, 27.12.24).—Air is introduced under pressure into the molten iron, through openings in the wall of the furnace or through refractory ducts which pass through the wall and terminate below the surface of the metal. Additional heat is not required, since the removal of sulphur and silicon which takes place is an exothermic reaction. The purified iron is drawn off, leaving the impurities behind as a slag, at the end of the process.

B. W. CLARKE.

Decarbonisation of metals, especially iron and steel. SOC. DES ACIÉRIES ET FORGES DE FIRMINY (F.P. 591,981, 13.3.24).—The metal is decarbonised by hydrogen saturated with steam at 40°, in vessels from which the air has been removed. The hydrogen is saturated by passing through warm water, then through a fine spray of water, and is finally heated to 100°. The carbon monoxide and dioxide and methane produced may be removed from the gas mixture, or a corresponding amount of gas allowed to escape into the air. The carbon content of the iron is reduced to less than 0·01 %.

B. W. CLARKE.

Manufacture of deoxidised iron or steel. W. TAFEL (G.P. 399,906, 19.6.23 and 409,347, 3.6.24).—Slag from weld iron or a slag of similar composition is spread on the ingot mould in a solid state before the metal is poured, and the molten metal mixed well therewith. The product is suitable for hot working and is stable at high temperatures. The method described prevents melting of the cast iron plates at the bottom of the mould.

B. W. CLARKE.

Iron blast furnace process. HALBERGERHÜTTE G.M.B.H. (G.P. 404,700, 16.11.23).—To ensure smoother running of a blast furnace, especially when a high temperature is required, oxygen and fuel in the ratio necessary for the formation of carbon monoxide are simultaneously blown into the furnace.

A. R. POWELL.

Desulphurisation of iron and steel. W. KROLL (G.P. 418,074, 12.3.22).—The molten metal is treated with a mixture of alkalis and strong reducing agents, such as calcium carbide, aluminium, magnesium, copper, and silicon, so that the material of the walls of the bath is not attacked by the alkali. The sulphur content of an iron may thus be reduced from 0·12% to 0·06%.

B. W. CLARKE.

Increasing the amount and phosphoric acid content of slag from the Thomas process. H. HILBERT (G.P. 418,102, 20.2.25).—The limestone or dolomite added in the converter process is replaced by calcium phosphate, forming a slag containing the phosphate in a readily soluble form, with no harmful effects on the properties of the steel.

B. W. CLARKE.

Refining the grain of steel. F. KRUPP A.-G., Assees. of H. SCHOTTKY (G.P. 418,124, 6.11.23).—Steel is heated to above the upper transformation temperature (Ac2), quenched rapidly in a bath of liquid, and subjected to a second heating to just above the upper transformation temperature, followed by a moderately rapid cooling. The product possesses exceptional strength and toughness.

B. W. CLARKE.

Electrolytic iron. Siemens u. Halske A.-G., Assees. of H. Gerdien (G.P. 418,139, 23.6.23).—Cast iron anodes with a carbon content of more than 3% are used, so that after dissolution of the iron, a skeleton of carbon still remains. The voltage should be less than 1 volt and the temperature at the bottom of the vessel should be as low as possible. B. W. Clarke.

Coating ferrous metals to make them rust-proof. C. F. Hendrick (E.P. 244,523, 18.9.24).—Ferrous metals are subjected to the heat of an intensely hot flame whereby superficial fusion is produced, and simultaneously a protective non-ferrous metal or alloy, of lower melting point than iron or steel, is fed in a finely divided state at a rate such that it becomes diffused in molten form on or in the fused ferrous surface. The protective metal is introduced either directly on to the ferrous surface or, in powdered form, is driven on by the gases of the flame-producing mechanism or by a separate air stream. Oxidation during the process may be prevented by the use of a deoxidising flux. L. M. Clark.

Electrodepositing nickel, cobalt, or nickel alloys. E. Potter. From Madsenell Corp. (E.P. 244,166–7, 11.9.24).—(A) Soft, highly malleable and ductile nickel deposits may be obtained from a bath containing 240 g. of nickel sulphate crystals, 20 g. of nickel chloride, and 40 g. of boric acid per litre of water if the bath is first neutralised with sodium hydroxide or nickel hydroxide and treated with chlorine to oxidise and precipitate the iron and copper impurities. The bath is filtered before use and 4 c.c. of 3% hydrogen peroxide per litre are added every 24 hrs. The anode consists of nickel or a nickel alloy containing more than 0·1% of oxygen and is obtained by adding nickel peroxide to, or passing air through, molten nickel until a black deposit forms on the surface of the metal. (B) Hard, but malleable and ductile nickel containing more or less iron is deposited from a bath containing 300 g. of nickel sulphate crystals, about 30 g. of nickel fluoride or nickel hydrogen fluoride, and a small amount of quinol per litre. The anode consists of nickel or a nickel alloy free from chromium and silicon but containing more than 0·5% C ; it is made by stirring molten nickel at 1500° in contact with carbon for 30 min., cooling nearly to the m.p., and casting quickly. A. R. Powell.

Electroplating. J. G. Swain, Assr. to Firestone Steel Products Co. (U.S.P. 1,565,683, 15.12.25. Appl., 12.11.23).—The article to be electroplated is treated in an electrolyte charged simultaneously from anodes of zinc and mercury. A. R. Powell.

Producing electrolytic copper. F. L. Antisell (U.S.P. 1,566,265, 22.12.25. Appl., 11.1.22).—Solutions containing copper are electrolysed in cycles, the current being passed through the solution for a predetermined fraction of each cycle. L. A. Coles.

Electroplating method. R. J. Shoemaker (U.S.P. 1,566,984, 22.12.25. Appl., 1.10.23).—In electroplating iron articles the surface is covered with a permanently adherent film of mercury and a metal capable of forming an amalgam with mercury, and then plated. M. Cook.

Electrolytic deposition of chromium. G. le Bris (F.P. 590,777, 1.12.24).—Chromium is deposited electrolytically from a bath containing chromic salts together with salts or acids having an oxidising action, using lead anodes, a current of 4—6 volts and 7—8 amp. per sq. dm., and a temperature of 35—40°. L. A. Coles.

Welding. C. H. Humphries, Assr. to Metals Protection Corp. (U.S.P. 1,563,748, 1.12.25. Appl., 31.8.25).—Iron or steel surfaces to be welded are treated with a solution of chromic acid and then allowed to dry before welding. Sputtering of the metal is thus prevented. T. S. Wheeler.

Recovering tin from ores. J. R. Stack, Assr. to Amer. Smelting and Refining Co. (U.S.P. 1,566,352, 22.12.25. Appl., 23.5.19).—Ores, concentrates, and by-products containing compounds of tin, iron, and other elements are crushed, heated in a closed receptacle to a temperature above the reducing point of tin and below the reducing point of iron, and subjected to the action of a gas which will reduce tin oxides. Tin so produced is removed, and the residue treated for the recovery of other metals. L. M. Clark.

Treating sulphide ores and concentrates. M. de Keyser (U.S.P. 1,566,379, 22.12.25. Appl., 21.7.22).—The material is roasted to produce sulphur dioxide which is then mixed with ozone and passed in contact with a catalyst. M. Cook.

Producing an alloy of copper. C. Kazemer (U.S.P. 1,566,406, 22.12.25. Appl., 11.6.25).—Copper is melted with glass and made into sheets which are reduced in thickness by pressure and re-melted with a mixture of silver and glass. This melt is formed into a sheet which is reduced in thickness and into a workable form. M. Cook.

Method of electrolytically producing aluminium. J. B. Railsback (U.S.P. 1,566,694, 22.12.25. Appl., 20.10.24).—The bath contains compounds of aluminium and a metal the oxide of which is not reducible by aluminium. M. Cook.

Treating ores. C. A. McCourt (U.S.P. 1,566,755' 22.12.25. Appl., 12.1.24).—In a continuous process for recovering the lighter metal values from slimes, an amalgamating solution is caused to flow through an electrolytic cell and streams of this solution are then diverted on to the slimes at separate points. The amalgamated lighter values are separated from the solution and the spent liquor is returned to the electrolytic cell to prepare it for use again in the process. L. M. Clark.

Apparatus for manufacturing metal dust. M. H. Newell, Assr. to Alloys Co. (U.S.P. 1,566,913, 22.12.25. Appl., 29.8.16).—In an apparatus for manufacturing metal dust by condensing vaporised

metal, a condensing chamber is provided with an aperture and means for retarding the inflow of air through the aperture, the chamber being otherwise substantially airtight. L. M. CLARK.

Heat treatment of grey iron castings. A. K. SCHAAP (E.P. 245,172, 1.7.24).—See U.S.P. 1,514,070; B., 1925, 44.

Deoxidation of ingot iron and steel. W. TAFEL (E.P. 245,486, 18.9.24).—See G.P. 399,906; preceding.

Treatment of oxidic raw materials. T. R. HAGLUND (U.S.P. 1,569,483, 12.1.26. Appl., 5.8.22). —See E.P. 232,549; B. 1925, 957.

Metal melting furnaces. L. HALL (E.P. 245,390, 24.12.24).

See also pages 145, **Platinum–ruthenium catalyst** (G.P. 418,868). 147, **Blast-furnace gas purification** (E.P. 244,372). 153, **Treatment of black liquor** (Can. P. 245,831). 156, **Precipitating tin** (E.P. 244,526); **Purifying zinc sulphate solutions** (Swed. P. 57,951). 158, **Abrasives** (G.P. 417,888). 164, **Electro-thermal process** (F.P. 592,045).

XI.—ELECTROTECHNICS.

High-frequency induction furnace. D. F. CAMPBELL (J. Iron and Steel Inst., 1925, **112**, 69—77). —See B., 1925, 812.

See also A., 1926, 104, **Action of visible light on electrodes** (AUDUBERT). 115, **Unipolarity phenomena in compressed powders** (TREY). 130, **Overvoltage** (MEUNIER); **Electrolysis of acid solution of copper sulphate** (REDMAN). 134, **Production of sulphuric acid by electrolysis** (SAXON). 147, **Electrolytic reduction of aldehydes** (SHIMA).

Electric-furnace melting of non-ferrous metals. TAMA.—See X.

PATENTS.

Galvanic cell of the copper oxide type. G. W. HEISE, Assr. to NAT. CARBON CO. (U.S.P. 1,563,980, 1.12.25. Appl., 27.5.24).—In a galvanic cell with a copper oxide depolariser and caustic soda as electrolyte, the depolariser is suspended in the electrolyte in a container lined with cellulose. This prevents any dissolved copper passing out into the cell where it would damage the zinc electrode. T. S. WHEELER.

Electrolysis of alkali chlorides. H. KLOPSTOCK (U.S.P. 1,565,943, 15.12.25. Appl., 25.5.25).—In the use of the mercury process the mercury is protected by an air-excluding fluid throughout its transfer by a propelling device from the amalgam-decomposition cell to the amalgam-formation cell. H. HOLMES.

Impregnated electrode for furnace work. H. A. LAVENE, Assr. to ACHESON GRAPHITE CO. (U.S.P. 1,566,409, 22.12.25. Appl., 5.2.23).—An electrode having in its pores a refractory compound formed by the interaction of two soluble compounds is claimed. M. COOK.

Electric furnace. A. GRAUEL (U.S.P. 1,566,855, 22.12.25. Appl., 30.4.23).—The furnace consists of a casing enclosing a water-jacket and a central closed heating chamber, the bottom portion of which is lined with an electrical insulating material. A bed of loose carbon particles is arranged within the lined portion of the chamber and electrodes are inserted through, and insulated from, the water-jacket walls. The carbon particles form a heat-radiating body directing heat rays on to the inner surface of the water-jacket. M. COOK.

Electro-thermal process. NORSKE AKTIESELSKAB FOR ELEKTROKEM. IND. (F.P. 592,045, 23.1.25. Conv., 8.2.24).—Electrodes having a higher electrical resistance than carbon, constructed, *e.g.*, of mixtures of carbon, material of high resistance, and binding material, are used in heating electric furnaces. The material to be heated is stacked around the electrodes, which may be provided with metal ribs to facilitate passage of the current. The electrodes may be constructed of the reacting material itself, *e.g.*, of mixtures of carbon, iron or zinc ores, and binding material, in which case they may be up to 2 m. in diameter. L. A. COLES.

Ozoniser. P. J. ARENA (F.P. 592,644, 8.11.24).—The apparatus generates a high-tension current, producing sparks which ozonise a current of air passing through the apparatus. L. A. COLES.

Apparatus for electrolysing fused salts of metals and recovering the metals and acid radicals. E. A. ASHCROFT (U.S.P. 1,569,606, 12.1.26. Appl., 6.2.24).—See E.P. 215,872; B., 1924, 602.

Diaphragms to be used in electric batteries. M. WILDERMAN (E.P. 245,252, 12.9.24).

See also pages 145, **Treating gases at high temperatures** (F.P. 593,298). 147, **Electrical gas purification** (E.P. 244,372); **Insulating oils** (G.P. 417,835). 147–8, **Dehydrating emulsions** (U.S.P. 1,565,992 and 1,565,997). 153, **Treatment of black liquor** (Can. P. 245,831). 158, **Abrasives** (G.P. 417,888). 159, **Cement and mortar** (E.P. 244,603). 170, **Purifying sugar juice** (F.P. 591,198).

XII.—FATS; OILS; WAXES.

Tung oil. W. NAGEL and J. GRÜSS (Z. angew. Chem., 1926, **39**, 10—13).—The earlier theories of the gelatinisation process of tung oil are briefly indicated, attention being mainly given to the views of Marcusson, Grün, and Wolff. The last-named's insistence on the absence of molecular polymerisation has been criticised by various workers on the ground of the uncertainty attaching to molecular weight determinations, and the present authors use the iodine value as their criterion. When methyl α- and β-elæostearates were heated at different temperatures for various lengths of time in an atmosphere of carbon dioxide, a limiting value of 80—90 for the iodine value was obtained, further heating inevitably producing destructive distillation. The iodine value

of methyl elæostearate is 172·5, so that the observed value indicates the formation of a dimeride. Similar treatment of tung oil yielded a limiting iodine value of 110—120 for a product just about to gelatinise. In this connexion it was found that 10% ethereal hydrogen chloride was an effective reagent for liquefying without depolymerising, freshly gelatinised tung oil. The iodine value of elæostearin being 173·3, the fall in this value corresponds to polymerisation to the extent of 60% of the original oil. Typical formulæ for the dimeride are given, all of which are characterised by the presence of a tetramethylene ring. By parallel experiments with amyl and glycol esters of elæostearic acid it was shown that the solidification of the dimeride is dependent on the nature of the alkyl group, since methyl and ethyl esters remain liquid, the amyl ester partially solidifies, whilst the glycol and glyceryl esters completely solidify on polymerisation. This is in harmony with the authors' views and previous work on shellac (cf. B., 1923, 1234 A), whereas according to Wolff the methyl ester must differ fundamentally from the glyceryl ester, a view not borne out by these experiments. S. S. WOOLF.

Detection and determination of coconut oil and milk fat in cacao butter. J. KUHLMANN and J. GROSSFELD (Z. angew. Chem., 1926, 39, 24—25).—The method is similar to that used by Bertram, Bos, and Verhagen (Z. Deuts. Oel- u. Fett-Ind., 1924, **44**, 445—447, 459—461) for the determination of coconut oil and milk fat in margarine, and furnishes two values: the "A" number, for the fatty acids the magnesium soaps of which are soluble and the silver soaps insoluble in water, and the "B" number for volatile fatty acids with soluble silver soaps. Both numbers are in c.c. of 0·1 N-acid (or 0·1N-silver nitrate and 0·1N-sodium hydroxide) expressed on 6·4 g. of fat. An examination of many pure cacao fats shows that the "A" number does not rise above 0·2 and is generally about 0·1, whereas that of pure coconut oil is 27·7 and of milk fat 6·7, the "B" numbers being 0·3, 2·75, and 33·4 respectively. Both values for mutton tallow, mowrah fat, sesamé oil, linseed oil, and whale oil are below 1·16; for palm kernel oil the "A" value is 16·53—16·76 and the "B" value 1·80—1·92. A special advantage of the method is that the "A" value practically represents the coconut oil content, and the "B" value the milk fat content, an "A" number of 1 c.c. corresponding to 3·6% of coconut oil. Exactly 20 g. of the fat and 30 g. of glycerol are heated with 8 c.c. of 75% potassium hydroxide solution in a tared flask until saponification is complete. Warm water is added to make 409 g., the temperature adjusted to 80°, and 103 c.c. of magnesium sulphate solution (150 g. $MgSO_4,7H_2O$ in 1 litre) at 80° are added with shaking, which is continued for 10 min. The liquid is cooled to 20°, kept for 5 min., and filtered. 200 c.c. of the filtrate are added to 20 g. of sodium nitrate and neutralised with 0·5N-sulphuric acid using phenolphthalein (1 drop), 25 c.c. of 0·2N-silver nitrate are added, and then water to 250 c.c. After shaking and keeping in water at 20° for 5 min. the mixture is filtered and the excess of silver nitrate in 200 c.c. of filtrate determined by Volhard's method. The difference between this and the blank is the "A" number. 200 c.c. of the filtrate from the magnesium soaps are neutralised with 0·5N-sulphuric acid, made up to 250 c.c. with water, and 2 g. of powdered silver nitrate added with shaking at 20°. It is then filtered and 200 c.c. of the filtrate are acidified with 50 c.c. of dilute sulphuric acid (2·6%) and distilled from a 500-c.c. flask. 200 c.c. of the distillate are titrated with 0·1N-sodium hydroxide solution and the result minus the number of c.c. for the blank is the "B" number. E. H. SHARPLES.

Free thiocyanogen and its application in volumetric analysis. New criterion for fats and oils. H. P. KAUFMANN (Arch. Pharm., 1925, **263**, 675—721).—The chemistry of thiocyanogen is exhaustively reviewed, with copious citation of the literature (cf. in particular, Bjerrum, "Die Rhodanide des Goldes und das freie Rhodan," Copenhagen, 1918; Söderbäck, A., 1920, i, 219); the pharmacological importance of thiocyanogen compounds is similarly discussed; and the following new observations and applications are described in detail. Free thiocyanogen, which can be prepared in various ways (cf. Söderbäck, *loc. cit.*, Kaufmann and co-workers, A., 1925, i, 1252, etc.), displaces iodine from iodides, but is displaced from thiocyanates by bromine. It takes part in addition and substitution reactions like a halogen, being less active than bromine but more active than iodine (cf. Kaufmann and co-workers, A., 1923, i, 765; 1924, i, 209, 835, 840). On these facts new titration methods, applicable to fats and oils, and yielding results not otherwise obtainable, are based. For this purpose the thiocyanogen is obtained as a solution in an indifferent solvent (by interaction of lead thiocyanate and bromine). The stability of the solution depends on the nature of the solvent, as this has an influence on the rate at which polymerisation and decomposition take place. The nature of these changes is discussed at length (cf. Lecher and co-workers, Ber., 1923, **56**, 1204). The strength of the solution decreases at first slowly, but later very rapidly. Thus, in pure carbon disulphide 7% disappears in 12 days, whereas in technical carbon disulphide over 90% is lost in the same time. Similar losses take place in chloroform, carbon tetrachloride, and ethylene dichloride, the last of these being itself attacked. Ether and methyl alcohol likewise undergo substitution, both very rapidly. Solutions in acetic acid (absolutely anhydrous) are, however, more stable, *i.e.*, there is no loss in 3 days, and such solutions, in 0·05N or 0·1N strength, are well adapted for volumetric work. A series of "rhodanometric" determinations of the iodine values of fats and oils has accordingly been made, using 100—150% excess of thiocyanogen, and determining the excess by means of potassium iodide (cf. A., 1924, i, 840). The results are calculated as if iodine were used so as to be directly comparable with the usual iodine values. In the list following these figures are denoted by R and I respectively, the iodine values proper being

determined either by the Hanus method or by Kaufmann's bromometric method (cf. A., 1925, ii, 555). The period of interaction is indicated in each case. Castor oil, 6—12 hrs., R, 81·6; I, 82·6. Olive oil, 5 hours, R, 76·6; I, 80·8. Arachis oil (three samples), 12—15 hours, R, 78·5, 70, 68·1; I, 87·1, 89·3, 88. Almond oil (two samples), 14 hours, R, 83·8, 85·2; I, 97·59, 97·9. Rape oil, 5 hours, R, 77·4; I, 105·9. Sesamé oil, 7 hours, R, 75·5; I, 108·9. The differences between the two sets of figures in some cases are due to the selective action of the thiocyanogen (cf. Kaufmann and Wolff, A., 1924, i, 835). The following figures were obtained for purified fatty acids. Oleic acid, R, 90·6; I, 90·6. Elaidic acid, R, 81·5; I, 81·6. Erucic acid, R, 73·7; I, 74·0. Brassidic acid, R, 73·2; I, 73·8. These results show that compounds with one ethylenic linkage give the same result by either method and that stereoisomerism has no effect. Stearolic and behenolic acids are not affected by the reagent. Linoleic acid (as glyceryl ester), however, gave the numbers: R, 82·5; I, 169·1, *i.e.*, the thiocyanogen attacks only one ethylenic linkage. From the results obtained the composition of various natural oils was calculated. Castor oil is thus found to contain 1·15% of linoleic triglyceride; olive oil has the composition 10·92% of glycerides of saturated acids, 84·2% of olein, 4·88% of linolein; in arachis oil (third sample) the proportions are 21·2%, 55·66%, and 23·14% respectively; and in almond oil (second sample) 1·5%, 83·73%, 14·77% respectively. Rape oil contains 33·14% of linolein, 10·4% of saturated glycerides, the remainder being those of erucic and rapinic acids. Sesamé oil consists of 38·84% of linolein, 48·61% of olein, and 12·55% of saturated compounds. These results compare favourably with those obtained by a laborious chemical separation, in the cases in which such has been carried out. W. A. SILVESTER.

Rapid method for determining reaction velocity in soap boiling. G. I. FINCH and A. KARIM (J.S.C.I., 1926, **45**, 35—36 T).—A sample of the reaction mixture is diluted to a 2—3% solution, 25 c.c. of which are saturated with sodium chloride and then titrated against standard acid with phenolphthalein as indicator. Near the end-point some light petroleum is added to break down the grains and after shaking the titration is completed. The total alkali is obtained by titrating a separate portion, using methyl-orange as indicator. The results are corrected for carbonate, if any is present. The difference of the two readings gives the combined alkali, from which, and the saponification value of the original oil, can be calculated the degree of saponification.

See also A., Feb., 1926, 122, **Viscosity of soap solutions** (CLARKE). 123, **System sodium stearate–water** (VON BUYAGH). 134, **Catalytic hydrogenation** (SCHMIDT).

Waxes of cottons of different origin. LECOMBER and PROBERT.—See V.

Identifying fatty ingredients in sized goods. LECOMBER and PROBERT.—See V.

PATENTS.

Extraction of oil from oil-bearing substances. A. W. SIZER (E.P. 244,557, 25.10.24).—The material, with or without preliminary heating and crushing, is forced through perforated vessels, *e.g.*, by a worm, and the oil is thus continuously removed, after which the material is treated with volatile solvent, to extract the remaining oil. D. G. HEWER.

Production of soaps. R. VIDAL (F.P. 594,146, 26.2.25).—Oils, fats, and waxes are treated with alkali hydroxides in the presence of aldehydes, such as furfuraldehyde and methylfurfuraldehyde. The soaps thus obtained are specially suitable for brightening sulphur dyes. A. GEAKE.

Grease from slaughterhouse offal (U.S.P. 1,567,014).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Isomerism of Guignet's green, a phenomenon of particle size. L. WÖHLER and J. DIERKSEN (Z. angew. Chem., 1926, 39, 13—16).—Guignet's green is a hydrated oxide of chromium, produced in practice by fusion of potassium dichromate with boric acid, and the failure of attempts to convert the dull olive-green precipitated hydroxide into the brilliant pigment by heating with water under pressure, supported the view that the compound $2Cr_2O_3,6B_2O_3$ is responsible for the brightness. It is now shown that the presence of boric acid is not essential to Guignet's green, for which a gel structure is suggested. Reduction in the amount of water in chromium hydroxide gels, with increase in particle size, is found to increase the brilliance of the product progressively. Boric acid, silicic acid, etc. are effective in producing the required flocculation. Chromium oxides of differing hydration and particle size were prepared both from the pure precipitated hydroxide and by various flocculation processes, and the vapour pressure–temperature curves for these are discussed; brilliant flocculated hydroxides show higher vapour pressure than dull non-flocculated hydroxides of identical water-content. *X*-Ray photographs reveal the absence of any lattice structure. S. S. WOOLF.

Fastness of pigments to light and its determination in natural and artificial light. H. WAGNER (Z. angew. Chem., 1925, **38**, 1191—1195).—In estimating the fastness of a dyestuff or pigment it is important to follow the course of its fading, and this may be done graphically by plotting time as abscissæ and the corresponding shade according to the Ostwald scale as ordinates. Curves of a similar nature are usually obtained by illuminating with sunlight or with a "Uviol" (mercury) lamp, but the latter is generally 5—30 times as intense in action. A better method is to plot the progress of illumination by means of Ostwald's triangular axes, omitting time. The nature of the binding medium may have a profound influence on the fastness of a pigment, and its refractive index should be as near as possible

to that of the pigment. The least favourable medium for fastness to light is an aqueous glue wash, and the most favourable an oil or concentrated dextrin medium in which the medium constitutes an optical bridge and allows the light to strike deeper into the colour. This influence of binding medium on the behaviour of pigments on illumination is also best expressed graphically by means of triangular coordinates. W. T. K. Braunholtz.

Heterochromatic photometry. C. Schaefer (Physikal. Z., 1925, **26**, 908—913).—Earlier work on the measurement of the brightness of pigments (B., 1925, 179) is extended, confirmed, and placed on a more accurate basis. R. A. Morton.

Testing the rust-protective power of paints. P. Herrmann (Korrosion u. Metallschutz, 1925, **1**, 80—84 ; Chem. Zentr., 1925, II., 2326).—The painted surface of the iron is covered with a layer of cotton wool saturated with a solution of an electrolyte and in contact with a calomel electrode, and the point at which the paint film has been perforated is ascertained by means of a galvanometer. With micaceous paints rust can sometimes be observed even when there is no deflection of the galvanometer.
A. R. Powell.

Tung oil. Nagel and Grüss.—See XII.

Patents.

Preparation of lakes from organic dyestuffs. L. D. Gimberg and M. H. Bailly (F.P. 582,506, 29.4.24).—An aqueous or alcoholic solution of an aniline salt is treated with an organic sulphonic acid, and a nitro- or aminoazo-compound is added to the mixture. After filtering off the organic acid the filtrate is treated with a solution of an aluminium salt and the precipitated lake is washed free from all soluble substances. It may then be mixed with pastes of aluminium hydroxide or silicic acid.
A. R. Powell.

Lakes (F.P. 590,464).—See IV.

Monoazo dyestuff (Swiss P. 109,706).—See IV.

Plastic masses (E.P. 241,858).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Spraying [rubber] latex with protective colloids. W. C. Davey (J.S.C.I., 1925, **44**, 515—517 t).—Experiments on the spraying of latex to powder form by means of protective colloids, using an experimental Krause machine, are described. Glue was found to be the most suitable substance. Compounded moulded articles were made from suitable powders by pressing and curing in plunger moulds. With vulcanised latex 3% of glue yielded a powdery product which could also be moulded to shape. The use of a sprayed glue–rubber powder mixture is suggested as a suitable means of incorporating glue in rubber mixings on the rolls, and results are shown indicating improved reinforcement. The uncured mixes tend to deteriorate during storage. When using glue it is advisable to have zinc oxide present in the mix in order to obtain the best results in regard to tensile strength.

Factors influencing plasticity of sole crêpe [rubber]. H. P. Stevens (Bull. Rubber Growers' Assoc., 1925, **7**, 716—722).—The spread of crêpe rubber soles is due to the plasticity induced by machining, which has been held to rupture the protein films surrounding the particles of the rubber hydrocarbon ; the behaviour of rambong (*Ficus elastica*) and *Castilloa elastica* rubbers, the particles of which have no skin, makes this hypothesis doubtful. The method and results of plastometer tests are described. S. I. Levy.

Alkali [rubber] reclaiming process. W. E. Stafford (India-rubber J., 1925, **71**, 59).—Reclaiming rubber by heating vulcanised waste with sodium hydroxide solution under pressure removes free sulphur and depolymerises a portion of the vulcanised material, reducing the coefficient of vulcanisation in this portion and rendering it soluble in chloroform. The resin content is not increased. S. I. Levy.

Patents

Drying [rubber] latex. P. C. Rushen. From General Rubber Co. (E.P. 244,619, 7.3.25).—Latex is fed, at a rate controlled by an automatic float device, into a shallow trough, in which rotates a cylindrical metal or other perforated screen. Heated air or flue-gases, forced through a narrow slit within the cylinder, detach the latex from the perforations and carry the material thus divided into a drying chamber ; the exit gases pass through a baffle tower.
S. I. Levy.

Incorporation of rubber in non-aqueous materials. J. M. Weiss (U.S.P. 1,563,410, 1.12.25. Appl., 18.12.23).—Rubber latex is mixed with a high-boiling non-aqueous solvent, such as paraffin wax, at 120°. The water present evaporates and an intimate mixture of rubber and the non-aqueous solvent is obtained. T. S. Wheeler.

Rubber latex compositions. [Distempers mixed with rubber latex.] T. M. Rigby (E.P. 245,540, 21.10.24).

Plastic masses (E.P. 241,858).—See V.

XV.—LEATHER; GLUE.

Preparation of hide powder for [tannin] analysis. L. Meunier, P. Chambard, and A. Jamet (J. Soc. Leather Trades Chem., 1925, **9**, 510—512 ; cf. B., 1925, 603).—Powders prepared from the same species of animal hide (*e.g.*, calf hide, fresh or salted) give approximately the same results. Pelt fleshings should not be used. The best material is the belly portion of ox-hide, which should be limed with lime only and after removing the flesh and grain surfaces by splitting, delimed with ammonium chloride or acetic acid, well washed, minced to a fine pulp, and left for 6 hrs. in a saturated solution of

carbon dioxide, to reduce the p_H to 4—4·5. The pulp is then drained, dehydrated with acetone, dried at 30°, and subdivided with a rasp or grater. The product contains about 0·1% of ash and has p_H 5·5.
D. WOODROFFE.

Report of the International Commission on the analysis of chrome leathers and chrome liquors. R. F. INNES (J. Soc. Leather Trades Chem., 1925, **9**, 508—509; cf. B., 1924, 25).—It is proposed to modify the peroxide method of chromium determination by extracting the fused peroxide-leather ash mixture with cold water containing 5 c.c. of saturated potassium permanganate solution and then filtering. Leather should be dried before extracting the grease with petroleum spirit. A further extraction with alcohol will give oxidised oils, and extraction with benzene will give the asphalt. The Woodroffe and Green method (B., 1922, 641 A) of determining alkali metals is recommended as provisional. Spent chrome liquors should be filtered after boiling with sodium peroxide, to remove insoluble calcium peroxide. The excess of sulphurous acid in spent thiosulphate liquors can be determined by titrating 25 c.c. of the filtered liquor with 0·1*N*-potassium dichromate To determine chromium in spent thiosulphate liquors, after determining the acidity the precipitated chromium hydroxide is filtered off, washed, dissolved in acid, and treated with sodium peroxide as usual. D. WOODROFFE.

Structure of gelatin gels. KRAEMER.—See A., Feb., 1926, 124.

Leather dyeing. I. SALT. **II.** MCCANDLISH and SALT.—See VI.

PATENTS.

Process for rapid tanning. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of F. MERKEL (G.P. 417,865, 13.1.22. Addn. to 414,867; cf. B., 1925, 858).—The chromium salts used in the process of the chief patent are replaced completely or partly by soluble aluminium salts of inorganic acids, principally aluminium chloride. Hydrolysis of the aluminium salts is diminished by concentrating the bath as far as possible. Neutral as well as basic aluminium chloride can be used. The tanning bath is of such concentration that the Al_2O_3 content is about the same as in the solid crystalline alum, or even higher concentrations may be used. A more rapid rate of tanning is thereby attained, and the hydrolysis and proteolysis, which occur in more dilute solutions, and adversely affect the tensile strength of the leather, are much decreased; further, crystallisation of aluminium salts on the skin, which occurs frequently when alum is used, is avoided. In this way soluble aluminium salts can be used for tanning heavy and medium leathers as well as for glacé and skin tanning. In many cases a combination with other tanning agents, *e.g.*, chromium salts, is advantageous.
A. B. MANNING.

Preparation of tanning agents. CHEM. FABR. WORMS A.-G. (G.P. 417,972, 11.7.20).—Two mols. of acetaldehyde are allowed to react with one mol. of a phenol in the presence of a normal sulphite, or on one mol. of an alkali phenoxide in the presence of the corresponding quantity of bisulphite, and acid or a metal salt is added to the product until it is neutral to Congo-red. The product, either before or after being acidified, may be mixed with natural tanning agents, or with glucose. As an example, commercial xylenol is heated with a solution of sodium sulphite and two mols. of acetaldehyde for several hours under pressure. The reaction product is acidified, the sulphurous acid boiled off, the residue neutralised with alkali, and treated with chromium sulphate. The dry product is only partially soluble in water, with separation of basic salts; the filtrate is acid to litmus and neutral to Congo-red, and precipitates gelatin from solution. On being heated it chars without melting. The soluble sodium salts of the condensation products are completely insoluble in benzol, chloroform, ether, and alcohol, and couple with aromatic diazo-compounds, so that skins treated with them can subsequently be dyed. A. B. MANNING.

XVI.—AGRICULTURE.

Steam and chemical soil disinfection with special reference to potato wart. N. R HUNT, F. G. O'DONNELL, and R. P. MARSHALL (J. Agric. Res., 1925, **31**, 301—364).—Potato wart can be eliminated from soil by sterilisation with steam under pressure or by chemical treatment. Fourteen of the twenty-two chemicals used produced wart-free plots during the season when treated and the two following seasons, but sodium fluoride inhibited growth of the plant completely and sulphur almost completely in all three seasons. The cost of treatment per acre and the water requirements are stated. The temperature changes occurring in soil heated under pressure with steam and the degree of penetration in various soils of various chemicals have been studied. A table showing the growth obtained in the seasons following treatment is given. H. J. CHANNON.

Colloidal silica and the efficiency of phosphates [as plant nutrients]. P. L. GILE and J. G. SMITH (J. Agric. Res., 1925, **31**, 247—260).—The addition of colloidal silica greatly increased the yield of millet in sand cultures when phosphorus was supplied as rock phosphate and slightly increased it when the source of phosphorus was superphosphate. On the other hand, the effect of a mixed gel containing iron, alumina, and silica, when rock phosphate was given, was to reduce the yield below that obtained with no phosphate. The amount of phosphoric acid taken up by the plants was proportional to the yield; and, in the presence of colloidal silica, an increased solubility of rock phosphate in the nutrient solution used was demonstrated. The silica, therefore, appears to act by bringing a greater quantity of phosphoric acid into solution and hence increasing the availability. The unfavourable action of the mixed gel was probably due to the production of soluble iron and aluminium by base exchange, with consequent diminution of soluble phosphate. The experiments are a preliminary to a study of the effect of the

colloidal material of soils on the efficiency of phosphates as plant nutrients. C. T. GIMINGHAM.

Reciprocal action of ground phosphorite and soils not having an acid reaction. A. N. LEBEDIANTZEV (Ann. sci. agron., 1924, 41, 320—329; Chem. Abstr., 1925, 19, 3340).—The biological activity of soils is of great importance in modifying the availability of phosphorite. Usually phosphorite was more active when added immediately before planting than when added to fallowed and subsequently cropped soil. Phosphorite is more sensitive to desiccation than basic slag or superphosphate, and it is consequently believed that the greater part of the phosphate of phosphorite passes into organic combination. The phosphate of phosphorite is thus believed to be inherently much more mobile under soil conditions than that of superphosphate or basic slag. A. A. ELDRIDGE.

Influence of aluminium, manganese, and iron salts on the growth of sugar cane, and their relation to the infertility of acid Island soils. W. T. MCGEORGE (Exp. Sta. Hawaiian Sugar Planters' Assoc. Bull., 1925, [49], 95 pp.; Chem. Abstr., 1925, 19, 3558).—Under the conditions described, aluminium salts exert a direct toxic action on sugar cane, whilst manganese salts have no effect. The resistance of the plant to aluminium toxicity is increased by increasing its phosphate or potash reserve. Phosphate applications are effective only over a short period, and heavy applications of potash are usually required. Lime gave no immediate response, but there was a greater residual stimulation of plant growth. A. A. ELDRIDGE.

Determination of nitrate. KÜRSCHNER and SCHARRER.—See VII.

PATENTS.

Stable ammonium bicarbonate (E.P. 244,645).—See VII.

Reducing offal to tankage (U.S.P. 1,567,014).—See XIX.

Colloidal metal solutions (G.P. 417,933).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Hydrogen-ion concentration as a basis of [sugar] refinery alkalinity control. A. A. BLOWSKI and A. L. HOLVEN (Ind. Eng. Chem., 1925, 17, 1263—1266).—Colour charts, consisting of dyed celluloid sheets representing the colours obtained with methyl-red, bromothymol-blue, and phenolphthalein between p_H 4·5 and 10, are used, these greatly simplifying the "spot test" method of operating. In the refining process the p_H is kept at approx. 7·0 by neutralising the acidity in the entering products and also that developed later in the refining operations. This control has resulted in more uniformly neutral conditions with the use of only half the amount of lime formerly added, thereby reducing the molasses production arising from the formation of melassigenic calcium salts. J. P. OGILVIE.

Influence of the filter-cloth on scum press work [in sugar refining]. J. HRUDA (Z. Zuckerind. Czechoslov., 1925, 50, 73—80, 81—87, 89—93, 113—118, 121—128).—Varying results in respect of rate of filtration, volume of sweetening-off water, clarity of filtrate, cleanliness of surface, durability, etc., were obtained in a series of factory tests made with carbonatation juices, using different kinds of cloth for the dressing of the filter-presses. Particularly good results were obtained with the marks "1105" (Herold) and PZZI (Slechta und Sohn). J. P. OGILVIE.

Solubility of sucrose in impure solutions. J. ROBART (Bull. Assoc. Chim. Sucr., 1925, 43, 128—132).—Working with beet molasses containing very melassigenic non-sugars, results were obtained showing that the solubility of sucrose is not affected by the presence of such substances. J. P. OGILVIE.

Jena sintered glass crucibles [in sugar determinations]. G. D. ELSDON (Analyst, 1926, 51, 30—31).—The crucibles of the size (1bG3/<7) are useful for weighing cuprous oxide in sugar determinations but must be selected by actual experience owing to individual differences. Efficient filtration should be obtainable at a trickle rate. Hot alkalis used in determinations by means of Fehling's solution cause a loss in weight of the crucible of the order 3 mg. D. G. HEWER.

Nitrogenous substances of beet molasses. E. PARISI (Annali Chim. Appl., 1925, 15, 555—568).—The possible uses of beet molasses are discussed and the following results given for the various forms in which nitrogen exists in Italian molasses: total, 2·11; protein, 0·11; ammoniacal, 0·02; amide, 0·04; nitric, 0·10; basic, 0·57; total monoamino-acids, 0·95; aminic, 0·45%, calculated on the weight of molasses. The nitrogen precipitated by Neuberg's reagent (mercuric acetate, sodium carbonate, and alcohol) amounts to 1·19%, composed of: aminic, 0·40; precipitated by normal lead acetate, 0·04; purine bases, trace; hexone bases, 0·16; total mono-amino-acids (by difference), 0·95. The nitrogen not precipitated by Neuberg's reagent is 1·02%, comprising: basic, 0·40; aminic, traces; slightly soluble in alcohol, 0·45; soluble in alcohol, 0·57. T. H. POPE.

Preparation of *l*-arabinose from mesquite gum. E. ANDERSON and L. SANDS (Ind. Eng. Chem., 1925, 17, 1257—1258).—Gum from the mesquite tree (*Prosopis juliflora*) is treated with 6 times its weight of 4% sulphuric acid, for 3 hrs. at 80°, the acid removed as barium sulphate, the filtrate concentrated, salts are precipitated by alcohol, and the alcoholic liquor is concentrated and allowed to crystallise. A yield of *l*-arabinose amounting to 27—36% of the gum can thus be obtained. J. P. OGILVIE.

Behaviour of starch components towards iodine, and their protective colloid action. SAMEC and KLEMEN.—See A., 1926, 22.

Hexahexosan and trihexosan. CASTAN and PICTET.—See A., 1926, 52.

Dihexosan and tetrahexosan. PICTET and SALZMANN.—See A., 1926, 52.

Sulphur-containing sugar. SUZUKI and MORI. —See A., 1926, 96.

Influence of aluminium, manganese, and iron salts on growth of sugar cane. McGEORGE.—See XVI.

PATENTS.

Activating decolorising char for use in refining sugar. C. B. DAVIS (U.S.P. 1,565,911, 15.12.25. Appl., 10.10.23).—A carbon-forming distillate of coal, non-volatile at normal temperatures, is incorporated with the bone char, which is then passed, out of contact with air, through a kiln at a red heat below 1000°. The product is cooled out of contact with air, washed with hot water, dried, and again passed through a kiln, out of contact with air, prior to use. H. HOLMES.

Purification of sugar juice by chlorination. TOKYO IMPERIAL INDUSTRIAL LABORATORY (F.P. 591,198, 24.12.24).—Sugar juice is treated with hypochlorous acid at 55°, or chlorine gas is led into the juice, and the free chlorine is afterwards removed by distillation *in vacuo* or neutralised by the addition of an alkali. Alternatively, free chlorine may be generated in the sugar juice by adding sodium chloride and subjecting the solution to electrolysis. A. J. HALL.

XVIII.—FERMENTATION INDUSTRIES.

Changes in the degree of dispersion of the proteins of barley during malting, mashing, and fermentation. W. WINDISCH, P. KOLBACH, and E. WENTZELL (Woch Brau., 1925, **42**, 287—288, 295—297, 303—306, 313—317, 323—325, 333—335).—At various stages in the malting of a barley and the production of beer from the same malt, the products were investigated to ascertain the proportions of soluble nitrogenous matter capable of passing through a series of ultra-filters having pores of approximately the following sizes: 500, 120—150, 20—30, 18, and 15 $\mu\mu$. It is concluded that during malting the finer particles increase in amount more than the coarser ones. Kilning reduces the amounts of both kinds. Boiling of the wort with hops increases the quantity of proteins which are only just soluble, at the expense of those of medium colloidal dimensions, but equilibrium between the particles of different sizes is restored during fermentation. The hops exert a temporary stabilising influence on the very fine protein particles. In a wort acidified until it has the normal reaction of beer, and then stored, great changes take place in the sizes of the protein particles, and a large amount of coagulation occurs. Unacidified wort undergoes similar changes, but not to the same extent. It is impossible to determine correctly the amount of nitrogenous matter assimilated by yeast during fermentation, owing to coagulation of proteins which takes place at the same time. When stored unfermented wort is boiled, there occurs an increase in the amount of nitrogenous matters having degrees of dispersion within the range of an ultra-filter, but when fermented wort is boiled such changes as occur concern chiefly the particles too large to be within this range. J. H. LANE.

Diastatic power of malt and barley. H. L. HIND, H. THREADGOLD, and C. W. B. ARNOLD (J. Inst. Brew., 1926, **32**, 26—32).—Owing to want of control of the reaction of the starch solution, preparations of soluble starch from different sources are liable to give discordant results when used for the determination of the diastatic power of malt or barley. Addition of a buffer mixture ensures a constant reaction and avoids the necessity of relying on washing or neutralisation to a given indicator. The chance of false results through accidental and unnoticed contamination of starch or distilled water is eliminated. In the case of malts concordant results are secured by using Sörensen's acetate buffer in quantity sufficient to give p_H 4·6. The variation in the diastatic activity of the enzymes of barley as determined by unbuffered starches is greater than is the case with malt. Concordant results for barley can only be obtained if, in addition to buffering to a constant p_H, every care is taken to use a perfectly soluble starch. It is proposed to modify the standard method for determination of diastatic power adopted by the Malt Analysis Committee of the Institute of Brewing by the use of 20 c.c. of the acetate buffer solution in each litre of starch solution and, after completion of the conversion, replacing the usual addition of 10 c. of 0·1N-sodium hydroxide by 20 c.c. to stop the diastatic action. C. RANKEN.

Determination of the colour of malts. V. BERMANN (Woch. Brau., 1925, **42**, 317—318).—Ostwald's doctrine of colour is explained at greater length than in the previous paper by the author and Laufer (B., 1925, 820). J. H. LANE.

Evaluation of hops by chemical and biological tests. J. S. FORD and A. TAIT (J. Inst. Brew., 1926, **32**, 19—25; cf. B., 1924, 568).—The preservative values of hops as obtained by a biological and by a chemical method are compared. The chemical values, $(\%\alpha+\beta/5)\times10$, were obtained by multiplying by 10 the amount of preservative substance estimated according to the empirical chemical method previously published by the authors. In the bacteriological test varying quantities of hopped wort were added to a sterile cold water malt extract of d 1·010 and p_H 5·5, and after re-sterilisation, inoculated with a vigorous 24-hrs. culture of "Bact. X." After being kept at 30° for 24 hrs., the point of suppression of the growth of the bacterium was noted. The authors consider that, using "Bact. X.," the conditions cannot be standardised so as to obtain satisfactory uniformity of development of this organism. Owing to the biological tests being made at different times with consecutive lots of the samples, a difficulty arose in

expressing the whole series on a common numerical basis. As the agreement between chemical and biological results was fairly satisfactory, the chemical values were used as a standard for reducing the various biological values to a common basis.

C. RANKEN.

Titration of the bitter substances of hops. L. HEINTZ (Woch Brau., 1925, **42**, 325).—In Seibriger's method for determining the bitter substance of hops (B., 1913, 442; 1914, 37) greater precision in the titration can be attained by adopting the method described by Lüers for the titration of coloured liquids (B., 1914, 761). Small square bottles of colourless glass may be used. 50 c.c. of the filtrate to be tested are run into each of two bottles. To the left-hand one 5 c.c. of phenolphthalein solution (in 96% alcohol) are added 1 c.c. at a time, and then 5 c.c. of 96% alcohol 0·5 c.c. at a time to clear the emulsion formed at first. Occasionally, 1 or 2 c.c. more alcohol may be required. Then 10 c.c. of alcohol are added to the liquid in the right-hand bottle. Two other bottles are placed behind, the left-hand one containing water and the right-hand one a phenolphthalein solution having the standard red colour. The titration is then carried out, equal volumes of alkali being added to the contents of each of the two front bottles, until on looking through the left-hand pair the colour appears the same as on looking through the right-hand pair.

J. H. LANE.

PATENTS.

Improving molasses as a nutrient medium for cultivation of aroma-producing bacteria. P. NITSCHE (Austr. P. 100,447, 26.7.24).—Molasses is heated with tannic acid or similar substances and then filtered, whereby basic substances, *e.g.*, betaine, and vegetable proteins injurious to aroma-producing bacteria are precipitated and removed.

A. J. HALL.

Utilising sulphite-cellulose waste liquor (Swed. P. 57,863).—See V.

XIX.—FOODS.

Filtration method of measuring sediment in milk. A. R. TANKARD (Analyst, 1926, **51**, 31).—Although filtration methods of measuring sediment in milk are useful as a rough sorting test and for giving ocular demonstration of the presence of impurities, none can be regarded as quantitative, and results by such methods cannot be made the basis of a prosecution under the Sale of Food and Drugs Act.

D. G. HEWER.

Composition of pectin. Determination of galacturonic acid in pectin. W. H. DORE (J. Amer. Chem. Soc., 1926, **48**, 232—236).—A method for the determination of galacturonic acid in pectin has been developed, based on measurement of the carbon dioxide evolved, when this acid is distilled with hydrochloric acid, according to the equation $CHO.(CHOH)_4.CO_2H \rightarrow C_5H_4O_2$ (furfuraldehyde) $+CO_2+3H_2O$ (cf. Nanji, Paton, and Ling, J.S.C.I., 1925, **44**, 253 T). The galacturonic anhydride contents of various materials, evaluated by this method, were as follows: "dearabanised" beet pectin (Ehrlich's "crude pectin" extracted with 70% alcohol), 39·66—44·48%; impure "cytopectic acid" from beet pulp (cf. Clayson, Norris, and Schryver, A., 1922, i, 206), 41·10—44·82%; pectic acid from beet pulp ("crude pectin" demethylated with sodium hydroxide, acidified, and precipitated with alcohol), 54·30%; and hydrolysed beet pectin (autoclaved with 1% sulphuric acid at 120°, neutralised with barium carbonate, and precipitated with alcohol), 59·20—61·26%. Commercial pectin gave irregular results, apparently due to the production of carbon dioxide by secondary reactions. Attempts to utilise the production of furfuraldehyde as a method for the determination of galacturonic acid by leaching out the "araban" portion of the pectin with 70% alcohol (cf. Ehrlich, Schwalbe's "Chemische Untersuchung pflanzlicher Rohstoffe," 1920, 94) indicated that two extractions with 70% alcohol reduced the soluble furfuraldehyde-yielding matter to a small and constant amount, but no method of calculating the galacturonic acid content from the furfuraldehyde yield of the alcohol-soluble portion could be devised. It is concluded that the ring complex containing the galacturonic acid (cf. Nanji, Paton, and Ling, *loc. cit.*) has, associated with it, furfuraldehyde-yielding substances extraneous to the ring, in varying proportions. F. G. WILLSON.

Chemical composition of the separative septa of the lemon. A. FICHERA (Annali Chim. Appl., 1925, **15**, 568—572).—These septa are composed of complex polysaccharides, the external ones of cellulose, galactosans, and glucosans, and the internal ones of cellulose and glucosans.

T. H. POPE.

Determination of lignin. PALOHEIMO.—See V.

Coconut oil and milk fat in cacao butter. KUHLMANN and GROSSFELD.—See XII.

PATENTS.

Apparatus for reducing the offal in slaughter houses to grease and tankage. H. P. TAYLOR, Assr. to P. B. BROKAMP (U.S.P. 1,567,014, 22.12.25. Appl., 5.8.22).—The offal is cooked in a tank having a screened opening in the side near the top for the entrance of steam, and a conduit from the part behind the screened opening leading to a receiving tank. so that the melted grease and part of the finely-divided protein may be floated off. The grease is forced through an outlet at the top of the receiving tank by introducing water below, and the protein is withdrawn from the bottom to an evaporating tank, steam-jacketed at the bottom, and the concentrated protein returned to the first tank. D. G. HEWER.

Manufacture of fertilisers and stock foods. A. WOOSNAM. From J. J. BERRIGAN (E.P. 245,285, 23.1.25).—See U.S.P. 1,524,233; B., 1925, 260.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of quinine. L. DAVID (Pharm. Ztg., 1926, 71, 26—28).—Quinine in cinchona bark is thus determined: to exactly 17 g. of 1% hydrochloric acid in a rubber-stoppered flask are added 1·7 g. of powdered bark, and the liquid is warmed for half-an-hour on the water-bath with frequent shaking. After keeping for half-an-hour with occasional shaking, the mixture is cooled, weighed, and made up to the original weight with 1% hydrochloric acid. It is then filtered, and the filter-paper squeezed out. To 10 g. of the filtrate are added, with shaking, 10 drops of 0·1*N*- sodium hydroxide and 10 c.c. of 20% disodium hydrogen phosphate, and the liquid is kept for half-an-hour, with stirring. The precipitate is filtered off, flask and precipitate are washed four times with 2% disodium hydrogen phosphate, the last traces of precipitate being wiped from the beaker with filter-paper, which is added to the precipitate. The latter is treated with 4 c.c. of 20% sodium hydroxide, shaken, 6 g. of sodium chloride are added, and the mixture is extracted twice with 15 c.c. of ether, shaking for 20 min. each time. After half-an-hour, 15 c.c. of the ethereal extract are evaporated, the residue is twice treated with 3 c.c. of ether and evaporated, and the residue dissoved in 20 c.c. of neutral alcohol and titrated with 0·1*N*- hydrochloric acid in the presence of lacmoid, using a micro-burette. B. FULLMAN.

Determination of santonin in santonin pastilles. R. EDER and W. SCHNEITER (Schweiz. Apoth.-Ztg., 1925, 63, 557—560; Chem. Zentr., 1926, I., 188—189).—Four pastilles are finely ground with seasand and shaken with chloroform (50 g.) for 5 min. After the addition of 20 drops of water, the vigorous shaking is continued for 0·5 min., after which 45 g. of extract are filtered into a tared flask; chloroform is removed by distillation and the flask with contents is dried at 100° for 1 hr. The residue should amount to 0·085—0·095 g., corresponding with a santonin content for one pastille of 0·024—0·026 g. The santonin can be identified by its m.p. (170°). L. M. CLARK.

Determination of alcohol and ethyl chloride in chloroform. C. NEWCOMB (Analyst, 1926, 51, 19—30).—In addition to the B.P. tests for anæsthetic chloroform the density should be determined before and after washing for 20 min. The chloroform is then refluxed for 2 hrs., during which time a volume of 10% caustic potash equal to that of the chloroform is continuously added. The chloroform is subsequently separated, washed for 20 min., filtered, and the density again determined. The difference between the first two densities plus 0·0005 for the water dissolved, less 0·0002 if from 0·05 to 0·5% of ethyl chloride is present, gives a measure of the "water-soluble substances reckoned as alcohol." The difference between the second and third densities gives a measure of the ethyl chloride, and the density of the residual chloroform should be within 0·0002 of 1·4747 at 27°. If more than 0·03% of ethyl carbonate or 0·3% of carbon tetrachloride are present the last density will indicate the fact (unless they should be present in equivalent amounts). The washing of the chloroform may be conveniently carried out in a separating funnel to which the wash water is supplied through a tube extending downwards to about ¾ in. above the stop-cock, and drawn out to a fine point; the water is drawn through by means of a vacuum pump and the outlet tube can be adjusted to different levels. About 10—20 c.c. of water are run through per minute. The most convenient method for determining the density was found to be by means of a Westphal balance. A preliminary calibration is essential, and a series of at least six determinations of densities at different temperatures is made, the results are plotted, and the best line of the right slope for the known change in density with temperature is drawn through them. D. G. HEWER.

Free thiocyanogen. KAUFMANN.—See XII.

Materials containing tartaric acid. GLASER.—See XVIII.

PATENTS.

Manufacture of *CC*-disubstituted compounds of barbituric acid and 4-dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone. P. PFEIFFER (E.P. 244,035, 3.4.25. Conv., 3.12.24. Addn. to 231,512; B., 1925, 942).—The methods previously applied to the preparation of compounds, in molecular proportions, of diethylbarbituric acid and 4-dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone are now applied to *iso*propylpropenylbarbituric acid (yielding a *compound*, m.p. 95—97°) and phenylethylbarbituric acid (*compound*, m.p. 129—131°). B. FULLMAN.

Medicinal preparation. J. K. LILLY, Assr. to E. LILLY AND CO. (U.S.P. 1,562,459, 24.11.25. Appl., 5.5.25).—A mixture of equal parts by weight of phenylcinchoninic acid and its hydriodide has superior analgesic properties to the hydriodide alone and does not produce iodism. T. S. WHEELER.

Morphine solution. Analgesic agent. C. W. HOOPER, Assr. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,563,086-7, 24.11.25. Appl., [A] 8.10.24, [B] 22.1.25).—(A) A solution of 0·5 g. of morphine, 2·5 g. of novocaine, about 50 g. of magnesium sulphate, and about 0·1 g. of sodium bisulphite in 100 c.c. of water is stable on storage. (B) A solution of about 50 g. of magnesium sulphate and 2·5 g. of novocaine in 100 c.c. of water has analgesic properties and is stable. T. S. WHEELER.

Determining the physiological value of remedial and medical substances [antirachitics]. T. F. ZUCKER, Assr. to UNIVERSITY PATENTS, INC. (U.S.P. 1,563,134, 24.11.25. Appl., 26.11.23).—The antirachitic strength of a substance can be determined by measuring its capacity for promoting the secretion of hydrochloric acid in the stomach. The measure of this quantity is obtained by feeding an animal for a standard time on a standard alkaline diet calculated to produce rickets, and then determining the p_H value of the fæces. A known quantity

of the antirachitic substance under test is then administered and the p_H value of the fæces again determined. T. S. Wheeler.

Styptics. C. R. Downs (U.S.P. 1,563,346, 1.12.25. Appl., 12.8.24).—A mixture of equal parts of phthalyl peroxide containing 40% of free phthalic acid and talc is a valuable styptic. T. S. Wheeler.

Manufacturing a perfume smelling like amber. A. Corti, Assr. to Chemical Works Flora (U.S.P. 1,565,796, 15.12.25. Appl., 26.1.23).—The use of nitrodibromobutyl-*m*-cresol methyl ether, m.p. 100°, having a smell of musk and a true smell of amber, is claimed. H. Holmes.

Preparation of dibromobarbituric acid. Parke Davis and Co., Assees. of A. M. Clover (Can. P. 245,806, 9.6.24).—Bromine is added to a saturated solution of barbituric acid at a temperature below 40°, dibromobarbituric acid is separated by filtration, and the mother liquor is saturated with barbituric acid and used again in the process. L. A. Coles.

Preparation of alcohols from ethers. E. Merck, Assee. of W. Krauss (G.P. 417,926, 8.3.24. Addn. to 407,487).—Alcohols are produced by treating the benzyl ether of the desired alcohol with hydrogen in the presence of noble metals as catalysts at the ordinary temperature, as described in the chief patent (cf. B., 1925, 474). For example, an alcoholic solution of amyl benzyl ether containing palladiumised charcoal, is shaken with hydrogen at 15° until 1 mol. is absorbed. After removing the catalyst, the product is diluted with water and extracted with ether, toluene and amyl alcohol being recovered from the extract by distillation. L. A. Coles.

Conversion of methyl sulphide into carbon tetrachloride and other chlorine compounds. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of A. Hallstein (G.P. 417,970, 1.2.24. Addn. to 416,603; B., 1926, 28).—Methyl sulphide practically free from mercaptans (*v.i.*) is converted into methyl chloride, sulphur chloride, and carbon tetrachloride, by saturating it with chlorine at the ordinary temperature and subsequently passing chlorine into it at 100—120°, preferably in the presence of iodine as catalyst. L. A. Coles.

Improving the odour of methyl sulphide. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of A. Hallstein (G.P. 418,129, 18.5.24).—Evil-smelling impurities present in methyl sulphide obtained as a by-product in the manufacture of sulphate-cellulose, are removed by treating the material with metal oxides or hydroxides in solution or in suspension, *e.g.*, with 40% potassium or sodium hydroxide, or with powdered lime, or with aqueous or gaseous ammonia. L. A. Coles.

Preparation of protocatechuic acid and pyrocatechol. Zellstoff-fabr. Waldhof, and P. Zickmann (G.P. 418,219, 31.7.23).—In the production of protocatechuic acid and pyrocatechol by treating lignin with fused alkali hydroxides, the addition of reducing agents which liberate nascent hydrogen, *e.g.*, zinc dust or aluminium powder, prevents oxidation of the protocatechuic acid to oxalic acid. The protocatechuic acid can be converted completely into pyrocatechol by splitting off carbon dioxide. For example, zinc dust is added to a melt containing lignin, potassium hydroxide, and water, at 250°; after cooling the product, dissolving it in water, and acidifying the solution with sulphuric acid, protocatechuic acid is separated by extraction with a solvent not miscible with water, and evaporation of the solvent. L. A. Coles.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Visible decomposition of silver halide grains by light. A. P. H. Trivelli and S. E. Sheppard (J. Physical Chem., 1925, **29**, 1568—1581).—The photochemical darkening of specially prepared silver bromide crystals is definitely oriented in the crystals in a manner dependent on their growth and structure (cf. B., 1923, 908 A). In the case of silver bromide grains from photographic emulsions the decomposition is largely irregular. Statistical measurements showed that in a given emulsion, the sensitivity of the grains as indicated by visible decomposition increases with increasing size. Visible sensitivity is relatively independent of sensitivity nuclei in the grains, a fact which explains the lack of parallelism between developable sensitivity and visible sensitivity of different emulsions. A tentative theory based on a hypothesis due to Weigert (A., 1918, ii, 50) is proposed. It is suggested that photochemical decomposition is oriented in the crystals according to gradients of ionic deformation or perturbation of electron orbits following certain directions of growth in the crystals. Since foreign inclusions, such as sensitivity nuclei and also reduced silver, will introduce deformation in the surrounding silver bromide, the visible decomposition becomes autocatalytically oriented. This explains the contrast in behaviour between specially prepared silver bromide crystals and emulsion grains, and also the effect of size. Considerations of diffraction and internal reflection of light in the crystals are inadequate to explain the distribution of visible blackening. Oxidising agents have a relatively small effect on visible sensitivity, compared with their effect on developable sensitivity. W. Clark.

See A., 1926, 109, **Photographic spectrophotometry** (Dorgelo). 113, **X-Ray analysis of mixed crystals of silver halides** (Wilsey).

Patents.

Screens for colour photography. H. Keller (E.P. 244,644, 16.5.25).—Screens are prepared by spraying a gelatin or other colloid layer with materials which dye the colloid only when it is unhardened, and which themselves harden the colloid. The colloid layer is sprayed successively with the reqnired dye solutions, for example, of dyes of the Pina-type,

or of most aniline dyes, the hardening agent being formalin, chrome alum, or some similar substance, until all the uncoloured spots are covered. An alternative method of dyeing is to sprinkle mixtures in powdered form of the dyes and hardener on the moistened colloid surface. The layer is finally washed to remove the dyes or solutions deposited on portions of the colloid which have been already dyed and hardened by a previous spraying.
W. Clark.

Sensitising kinematograph films for positive copies by means of solutions of dichromate. A. Weingarten (U.S.P. 1,564,161, 1.12.25. Appl., 12.1.25).—Films are sensitised by bathing the colloid in a solution of potassium dichromate, potassium ferrocyanide, and potassium bromide.
W. Clark.

XXII.—EXPLOSIVES; MATCHES.

Stability of explosives as an additive property. M. Tonegutti (Annali Chim. Appl., 1925, 15, 542—547).—Measurements made by the Taliani method (B., 1921, 562 A) show that the stabilities of ballistites of different composition correspond with those calculated from the proportions of the component nitroglycerin and collodion cotton (cf. Lenze and Pleus, B., 1920, 281 A, 468 A; De Bruin, 1921, 903 A; Brunswig, "Explosivstoffe," 1923, 172). Analogous results are furnished by powders of the cordite type.
T. H. Pope.

Reaction of nitroglycerin powders to the Angeli test in relation to the stability to heat. M. Tonegutti (Annali Chim. Appl., 1925, 15, 548—554).—The results obtained on applying Angeli's acidity test (B., 1918, 608 A) to acid and neutral nitroglycerin powders show that with ordinary ballistites there exists a close relationship between the acidity and the stability towards heat shown by the Taliani test, but not by the Abel test. With powders of the cordite type, on the other hand, the acid reaction corresponds with a low Abel stability but not with the results of the other stability tests. With such powders only the Angeli and Abel tests furnish results sufficiently definite to indicate the state of preservation of the explosive. Other stability tests, especially the silvered vessel test, serve moderately well to determine the various degrees of stability exhibited by cordites of different types in relation to their different chemical compositions, more particularly to their content of nitroglycerin and vaseline and to the presence or absence of stabiliser.
T. H. Pope.

XXIII.—SANITATION; WATER PURIFICATION.

Sensitivity of the *o*-tolidine and starch–iodide tests for free chlorine [in water]. A. M. Buswell and C. S. Boruff (J. Amer. Water Works Assoc., 1925, 14, 384—405).—Using varying quantities of carefully standardised chlorine water and distilled water chlorinated but free from excess chlorine for dilution purposes, colours were obtained with *o*-tolidine solution which checked Ellms and Hauser's dichromate standards (cf. B., 1913, 1125) over the range 0·02—0·2 p.p.m. of chlorine. In the *o*-tolidine method iron when present as the chloride or nitrate above 1 p.p.m. gives a yellow solution and interferes with quantitative determinations of excess chlorine; nitrites in low concentration do not interfere because they are oxidised by the chlorine added and the compounds formed have no effect on the *o*-tolidine reagent. Water more basic than p_H 8 should be treated with acid to bring it within the range p_H 3—8; sensitivity is impaired at low temperatures. The starch–iodide test is affected by small amounts of iron and nitrites in low concentration; manganese also interferes; the hydrogen-ion concentration is an important factor, the most sensitive concentration being that given by 2 c.c. of concentrated hydrochloric acid in 50 c.c. of water. The starch–iodide test is more sensitive at low temperatures. At ordinary temperatures the two tests are equally sensitive and each gives a positive test for 0·005 p.p.m. of excess chlorine; neither is interfered with by the presence of oxidised organic compounds in solution. The authors recommend the *o*-tolidine test for excess chlorine in water, using Ellms and Hauser's standards in half portions in order that 50-c.c. Nessler tubes may be employed. Where the temperature is low the starch–iodide test is proposed, as the use of the *o*-tolidine method with the regular standards would lead to over-dosing, with consequent odours and tastes.
W. T. Lockett.

Determination of the chlorine ion in water. H. W. van Urk (Pharm. Weekblad, 1925, 62, 1338—1343 also Z. anal. Chem., 1925, 67, 281—288).—The Dutch Codex specifies titration with silver nitrate in presence of magnesium oxide, using potassium chromate as indicator. The concentration of chromate should be between $0{\cdot}07 \times 10^{-2}$ and $1{\cdot}5 \times 10^{-2}$ *M*. Only if the water is alkaline towards tropæolin-O should it be acidified before addition of magnesium oxide; the latter is not the most suitable substance, and the use of sodium hydrogen carbonate is recommended. The author's conclusions are criticised in an added note by N. Schoorl.
S. I. Levy.

Micro-determination of oxygen content of water. Risch.—See A., Feb., 1926, 140.

Patents.

Treatment of natural base-exchange zeolite-like materials. E. B. Higgins (E.P. 244,506, 10.9.24).—Clay-like impurities are rapidly separated from a base-exchange material for water-softening, *e.g.*, glauconite, by subjecting it to a peptising and hydraulic grading process in one or more columns of upwardly-flowing liquid, *e.g.*, a faintly alkaline solution (0·5—1 lb. of caustic soda per ton) or a solution of common salt. The speed of the column is regulated so that the finer solid particles flow upward out of the apparatus, and the coarser ones settle to the bottom, and the speed of the liquid increases in succeeding columns.
D. G. Hewer.

Utilising refuse (E.P. 244,358).—See VIII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 19, 1926.

I.—GENERAL; PLANT; MACHINERY.

Filtration. J. W. Hinchley, S. G. M. Ure, and B. W. Clarke (J.S.C.I., 1926, **45**, 1—8 T).—The rate of flow of a liquid through a filter-cake is assumed to be proportional to the pressure of filtration, and inversely proportional to the resistance to flow, which is made up of the initial resistance due to the cloth, press, etc., and the resistance of the cake as it is built up. An expression is derived $T = BW + A^1W^2$, where T = time of filtration and W=weight of filtrate passing through 1 sq. in. of cake. The value of B depends on the initial resistance and should be constant with the same filtering medium; the value of A^1 should increase directly with the thickness of the cake. Experiments with calcium carbonate and barium phosphate in a small filter-press confirm this. Experiments with a vacuum filtration apparatus show that the pressure, the thickness of the cake, and the composition of the prefilt have no appreciable effect on the density of the cake, as measured by the percentage of voids present, with precipitates consisting of rigid particles; in the filter-press, however, an increased pressure produces an increase of density in the cake.

B. W. Clarke.

High-precision fractional distillation in the laboratory. W. A. Peters, jun., and T. Baker (Ind. Eng. Chem., 1926, **18**, 69—72).—Separations of low-boiling liquids are effected in a pyrex glass system, in which the distilling flask (500—1000 c.c.) is connected to a column, 12·5 mm. inside diameter at the base, with no constrictions at top or bottom, and with a filling of glass rings made by cutting soft glass tubing of 5 mm. external diameter into 5 mm. lengths. A convenient height for the column is 1·5 m., and it is jacketed with a single tube and prolonged at the top as a dephlegmator (about 20 cm. long), by means of which a definite controlled amount of reflux is supplied to the column. The supply of cooling water to the dephlegmator is regulated and also the level. The thermometer is placed in the vapour above the dephlegmator, and the side tube passes through a condenser, as small as may be, to prevent unnecessary hold up of condensate. Variations in sizes of the parts of the apparatus are necessary according to the size of the samples being dealt with. For liquids of high b.p. rather more elaboration is necessary, *e.g.*, a winding of 0·016 in. nichrome resistance wire may be spaced 3 or 4 times to the inch around the column jacket, and a rheostat used to adjust the heat input to radiation loss etc. Curves are given for mixtures of acetone–ethyl alcohol–water–*n*-butyl alcohol, and acetone–water–*n*-butyl alcohol, and the type of column described has been found useful for the separation of organic isomerides.

D. G. Hewer.

Use of methyl salicylate in a flow-meter. R. H. K. Foster (Ind. Eng. Chem., 1926, **18**, 82).—Methyl salicylate (oil of wintergreen) is suitable as a flow-meter liquid since it is not affected by grease films in the tube; it has a very low vapour pressure and rate of evaporation, is easy to fill into the apparatus, and air bubbles are easily removable. It dissolves rubber, so that a glass stopcock should be used, for which soap is a suitable lubricant.

D. G. Hewer.

Theory of sampling. Vallery (Ann. Falsif., 1925, **18**, 597—605).—Where a bulk is made up of less than 200 lots, the number which ought to be sampled is greater than is usually recognised. Of 20 lots, 14 ought to be sampled; of 100 lots, 21 ought to be sampled. When the bulk exceeds 250 lots, the number which ought to be sampled is almost constant, being 22—25 lots. When every lot of a bulk must pass a certain test it is very necessary that various different mixtures of the lots chosen for sampling should be analysed. G. T. Peard.

Application of micrurgy to colloid chemistry. E. A. Hauser (Kolloid-Z., 1926, **38**, 76—80).—A description of the apparatus used for microdissection or "micrurgy," and of the applications of this method of investigation to colloids, especially of rubber. N. H. Hartshorne.

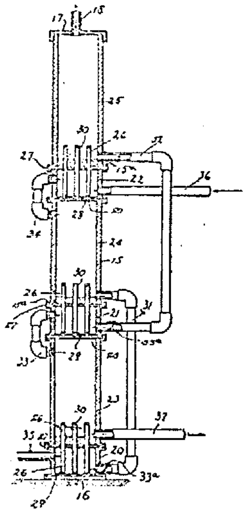
Photometric methods for studying suspensions of dusts, fumes, and smokes. Drinker, Thomson, and Finn.—See XXIII.

Patents.

Feeding solids and semi-solids to chemical apparatus. G. A. Berry (U.S.P. 1,565,249, 15.12.25. Appl., 17.3.20). — An apparatus for performing chemical reactions in which vapour is evolved and solid material is added during the reaction, comprises a closed tank fitted with a vapour pipe leading to a condenser from which a separate pipe returns the condensed liquid to the reaction vessel. The solid is added in a finely-divided form to the liquid flowing back to the vessel. If the reaction does not give rise to the evolution of vapour, a portion of the liquor is continuously withdrawn from the reaction vessel and pumped back to it, the solid being added to the returning liquid. The apparatus is suitable for use in reducing nitro-compounds by finely-divided iron.

T. S. Wheeler.

Absorption tower. D. L. NEWTON (U.S.P. 1,567,456, 29.12.25. Appl., 7.8.22).—The gas to be absorbed is admitted under pressure through the pipe, 35, at the base of the tower and together with the liquid passes upwards through the tubes, 30, through the various diaphragms. The gas alone passes upwards through the by-passes, 33 and 34, and is finally exhausted at the top, 18, while the liquid alone is supplied through the pipe, 36, and passes downwards through by-passes, 32, 31, which are provided with non-return valves, 33a, to prevent passage of gas.
B. M. VENABLES.



Filter for reclaiming crank-case oil and other mineral oils and animal fats. J. V. APABLASA, Assr. to C. C. APABLASA (U.S.P. 1,567,989, 29.12.25. Appl., 18.10.24).—At the bottom of a container a thick pad of felt rests on a wire screen supported by a perforated plate resting on a cover. The material may be preheated before entering the container, which contains an adjustable rotary agitator.
H. MOORE.

Filtering device. J. V. APABLASA (U.S.P. 1,567,990, 29.12.25. Appl., 11.3.25).—A detachable cover is secured to the bottom of a filter container, and supports rigid, perforated top and bottom filter plates, between which are other filtering elements. The perforations in the top plate correspond with those in the bottom plate. The perforations of the top plate contain filtering sand, and a rotating agitator in the container serves to clean the top plate.
H. MOORE.

Refrigerating apparatus. E. THOMSON (U.S.P. 1,568,102, 5.1.26. Appl., 28.7.23).—A compressor with a spring-supported piston, giving reduced delivery as the back pressure increases, draws the vapour of a refrigerant from the evaporator and compresses it into a condenser having a number of metallic fins and a chimney through which the air circulation increases with rising condenser temperature. From the condenser the liquid refrigerant expands through an "automatic intermittent" valve into an evaporator, or refrigerator proper, which is surrounded by such an amount of water that a layer of ice of sufficient thickness to serve as a thermal barrier to cover rest periods is formed.
B. M. VENABLES.

Continuous distillation. F. M. HESS (U.S.P. 1,568,157, 5.1.26. Appl., 29.12.21).—The material to be distilled flows by gravity through a series of vapour-boxes through which a heating agent flows in the reverse direction, the vapour from one of the vapour-boxes is passed through a higher vapour-box, and the liquid residue from the lowest vapour-box is also passed through a higher vapour-box. In all cases the heat exchanges are effected without direct contact of the materials. B. M. VENABLES.

Fractionating liquid mixtures. E. MERCK (F.P. 595,821, 26.3.25. Conv., 9.12.24).—An azeotropic system, formed by adding a suitable liquid to the mixture, is distilled under excess pressure. Thus alcohol and water may be separated by addition of benzene, and distillation. J. S. G. THOMAS.

Distillation of liquids under diminished pressure. G. DU BOIS (G.P. 420,235, 23.9.23).—The vapours generated in the still are passed through a rectifying column or through a series of chambers to enrich them in the most volatile constituent, and are then sucked by means of a vapour injector into a heat interchange apparatus or into a heating coil of the same still, where they are condensed. The injector vapour is obtained from a heater connected with the still, by means of a suction pump, and the composition of this vapour is brought into equilibrium with that obtained from the still by means of a tube joining the still and heater. A. R. POWELL.

Protection of metallic surfaces [from deposition of scale]. G. C. FREEMAN, Assr. to ANTISCALE CORP. (U.S.P. 1,568,728, 5.1.26. Appl., 13.4.25).—A metallic body is protected against deposition of scale from a fluid by connecting a source of electric current to spaced points in the body in such a manner that the current from the external source flows in the same direction as the "inherent current" in the body. B. M. VENABLES.

Ball grinding mill. P. T. LINDHARD, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,568,752, 5.1.26. Appl., 20.9.24).—A ball mill is provided with series of tubular screens arranged around and outside the grinding drum, parallel with the axis; material is admitted from the drum to the screens at one end and oversize returns to the grinding drum from the screens at the other end through a collecting tube, which is common to several screens and is bent inwards to return the material to the central part of the drum. B. M. VENABLES.

Apparatus for electrical precipitation of suspended material from gases. G. H. HORNE, Assr. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,569,492, 12.1.26. Appl., 13.4.25).—A collecting electrode is formed of semi-conducting material in which horizontal metallic members are embedded. A discharge electrode system comprising a number of vertical members of relatively small cross-section is arranged near the collecting electrode.
J. S. G. THOMAS.

Apparatus for electrical separation of suspended material from gases. E. ANDERSON, Assr. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,569,529, 12.1.26. Appl., 13.4.25).—A collecting electrode for electrical precipitators comprises a

body of high-resistance material, and an earthed metallic supporting and earthing frame in contact with but wholly outside the body, so that continuous electrical connexion is established between every part of the collecting electrode and the supporting and earthing frame through the high-resistance material of the electrode. J. S. G. Thomas.

Apparatus for electrical precipitation of suspended material from gases. F. H. Viets, Assr. to Internat. Precipitation Co. (U.S.P. 1,569,595, 12.1.26. Appl., 13.4.25).—A collecting electrode for use in electrical precipitators comprises an integral plate of semi-conducting material having a metallic supporting member extending along its upper edge, and metallic reinforcing and conducting members extending within and substantially throughout the whole height of the plate and secured at their upper ends to the supporting member.
J. S. G. Thomas.

Apparatus for electrical precipitation of suspended material from gases. C. H. Weiskopf, Assr. to Internat. Precipitation Co. (U.S.P. 1,569,597, 12.1.26. Appl., 13.4.25).—The discharge electrode system comprises parallel members of relatively small cross-section, whilst the collecting electrode system comprises a number of plate sections of semi-conducting material, metallic reinforcing and conducting members extending parallel to the discharge electrode system being embedded in each section. Conducting and reinforcing members in adjacent sections engage one another at their ends so as to provide continuous conduction and reinforcement for the collecting electrode. J. S. G. Thomas.

Electrical precipitation of suspended particles from gases. Siemens-Schuckertwerke G.m.b.H., Assees. of R. Heinrich (G.P. 421,151, 4.9.23).—The gas to be treated passes successively through two fields, having discharge and precipitating electrodes arranged so that the directions of the fields are reversed relatively to one another. For this purpose an alternating field of which the tension varies from the maximum to zero or approximately zero is established between the two fields.
J. S. G. Thomas.

Treatment of gases containing dust and smoke, prior to purification. Elektrische Gasreinigungs-G.m.b.H., and H. Rohmann (G.P. 421,259, 20.8.22).—The formation of electrically-charged flakes is effected under the influence of a brush discharge between electrodes which are cleaned mechanically, so that the flakes are carried forward in the gas stream and the process of agglomeration is completed. J. S. G. Thomas.

Preparation of colloids by condensation from molecular solutions. Maschinenbau-Anstalt Humboldt (G.P. 416,062, 17.6.21).—The colloid required is produced in a Plauson colloid mill or similar apparatus and is then washed and concentrated in a Plauson ultra-filter press. Examples are colloidal silver, copper oleate, tricalcium phosphate, and nickel sulphide. C. Irwin.

Preparation of very finely-divided solids. Maschinenbau-Anstalt Humboldt (G.P. 419,758, 7.1.23).—Solid substances, such as the more volatile metals, or compounds, such as naphthalene, which are capable of volatilisation, are evaporated and the vapour is passed into a Plauson colloid mill, together with a suitable gas which acts as a cooling and dispersing medium. A second dispersing agent may also be added, and the process is preferably worked under reduced pressure. In some cases chemical change may be effected, *e.g.*, zinc vapour may be oxidised to prôduce a preparation similar to lithopone.
C. Irwin.

Removing part of the contents of autoclaves [while working]. Farbw. vorm. Meister, Lucius, u. Brüning (G.P. 419,063, 27.6.22).—An airtight hollow element is charged with the gas or vapour present in the autoclave and brought to the same pressure and temperature as the latter. Connexion is then made with the autoclave, the hollow element is filled with the contents, which it is desired to remove, the connexion closed again, and the charge removed as desired. C. Irwin.

Intimately mixing liquids in mixers of the circulating pump or turbine type. Chem. Fabr. Griesheim-Elektron, Assees. of F. Sander and E. Kayser (G.P. 419,812, 21.2.23).—The liquids are mixed in a closed vessel with rotating paddles perforated close to the supporting shaft. The vessel is provided with a circulating tube which may be built out in the form of a heating or cooling tube or may be totally enclosed in the vessel by being built along the ribs or paddles. Part of the liquid passes along this tube from the region of highest pressure of liquid in the vessel and re-enters the main liquid through an adjustable valve in the region of lowest pressure. By the use of this apparatus a 93·5% conversion of sodium carbonate into hydroxide may be made by the addition of slaked lime to a 10% solution of sodium carbonate. A. R. Powell.

Non-rusting connexions for wooden vessels and apparatus to contain acid liquors. F. Hackel (G.P. 420,236, 8.2.25. Conv., 11.2.24).—Instead of joining with iron bolts, screws, or bands, the wooden members of vessels and apparatus designed to work with acid liquors, lead bosses, and clamps are cast into previously prepared channels in the wood so that the greater part of the lead is hidden. Corner pieces are joined together with lead hooks and bolts and wooden pulleys are joined together and fixed to steel shafting with rectangular lead bosses which are provided with lead wedges to grip the shafting. In making lead-lined tanks the lead lining is fastened tightly to the wooden walls by causing part of the lead to flow into a series of previously prepared holes in the wood. For stronger joints or for joining iron to wood the lead connexions may be reinforced with iron cores.
A. R. Powell.

Recovery of volatile compounds from reaction mixtures. Chem. Fabr. Griesheim-Elektron,

Assces. of H. SPECKETER, F. SANDER, and E. KAYSER. (G.P. 420,339, 8.6.22).—The reaction mixture is treated with acids, alkalis, or salts, which raise its boiling point, and the mixture is treated with steam, preferably by allowing the liquid to flow downwards through a column against a counter-current of steam, so that the temperature is always below the b.p. of the liquid. A. R. POWELL.

Solidifying liquid colloids. A.-G. FÜR CHEM. PROD. VORM. H. SCHEIDEMANDEL, A. OBERSOHN, W. WACHTEL, and D. SAKOM (G.P. 420,637, 30.3.23. Addn. to 418,865).—In a process for solidifying liquid colloids carried out in accordance with the main patent (cf. U.S.P. 1,534,729; B., 1925, 560), the gaseous cooling medium is constrained to flow along the walls of the chamber over which trickles continuously a liquid, preferably one in which the drops of colloid are soluble. The drops are deflected as much as possible by the walls of the chamber. J. S. G. THOMAS.

Recovering finely divided solid material from solutions or suspensions. L. HONIGMANN (G.P. 421,565, 13.10.23).—The solution or suspension is atomised in a circulating current of superheated steam, and the precipitated solid material and the excess of steam are discharged. This excess steam is then used as heating medium in a second circuit at lower temperature and pressure, and the excess from the second circuit is used in a third circuit, and so on. The plant comprises a series of heat interchangers, *e.g.*, plate heaters, of which the heat-absorbing part serves to form an atomising chamber, whilst the other part, from which heat is radiated, is in contact with the stream of excess steam. J. S. G. THOMAS.

Recovery of vapours from gases. R. SCHEUBLE (Austr. P. 101,042, 14.6.24).—Chlorine derivatives of naphthalene are used as absorption medium, preferably those obtained by direct chlorination of technical products. C. IRWIN.

Filtration of water or liquid. R. H. L. PENNELL (U.S.P. 1,570,523, 19.1.26. Appl., 24.4.22). —See E.P. 185,798; B., 1922, 874 A.

Apparatus for filtering dust-laden gases. T. THOMSON and N. NISBET (U.S.P. 1,570,869, 26.1.26. Appl., 12.9.25).—See E.P. 243,117; B., 1926, 80.

Resolving emulsions. E. E. AYRES, JUN., Assr. to SHARPLES SPECIALTY CO. (U.S.P. 1,570,987, 26.1.26. Appl., 2.4.21).—See E.P. 180,447; B., 1922, 580 A.

Gas-fired furnaces. ASKANIA-WERKE A.-G. (E.P. 234,792, 25.4.25. Conv., 31.5.24).

[Regulating feed to] pulverising mills. O. Y. IMRAY. From RAYMOND BROS. IMPACT PULVERIZER CO. (E.P. 245,850, 16.10.24).

Acid-proof and like containers. A. KELLY (E.P. 245,867, 23.10.24).

[Lifting device for bowls of] centrifugal separators. A. C. BAMFORD (E.P. 245,905, 10.12.24).

Roasting kilns (E.P. 245,348).—See X.

Heating processes and apparatus utilising atomic hydrogen (E.P. 237,898).—See XI.

Determining hydrogen-ion concentration of solutions (G.P. 399,410 and 400,273).—See XVI.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Coal dust explosions. Effect of release of pressure on their development. H. P. GREENWALD and R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 14, 1925. 12 pp.).—Preliminary experiments were carried out in the $7\frac{1}{2}$ ft. diameter gallery at Eskmeals to determine to what extent the development of a coal dust explosion was aided or retarded by release of pressure either at the origin of the explosion or at places along its path as at openings to galleries or drifts. It was found that the inception of a coal dust explosion was more favoured by prevention of release behind the point of ignition than by partial or complete release. When the openings were ahead of the flame its velocity was increased, and when they were behind the flame its velocity was decreased. The nearer the means of release of pressure were to the origin of the explosion the greater their effect. S. BINNING.

Stone dust as a preventive of coal dust explosions. G. S. RICE and R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 13, 1925. 15 pp.). —According to tests by the U.S. Bureau of Mines American coal dusts required more stone dust to render them incapable of transmitting a flame than apparently similar English coal dusts required when tested at Eskmeals. These differences were investigated by comparative tests of an American coal dust at Eskmeals and of an English coal dust (Altofts Silkstone) at Pittsburgh, and it was found that the differences were due to the American method of testing being more stringent than that in force at Eskmeals and not to any marked difference in the flame-propagating qualities of the two dusts. The greater stringency of the American tests was due to the use of a more intense source of ignition, to the large cross-sectional area of the roadway, containing the dust, and to the mode of distributing the coal dust, but in these respects the American test imitates conditions to be found in actual mining practice in America and is therefore justified. The American and English investigators disagree as to the effect of small amounts of combustible gas on the propagation of flame by mixtures of coal dust and stone dust and so far no explanation has been found. A possible explanation is the presence in the natural gas used in America of higher paraffins which have lower inflammabilities and lower ignition temperatures than the methane used at Eskmeals. S. BINNING.

Ignition of firedamp. H. F. COWARD and R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 8, 1925. 25 pp.).—The conditions under which firedamp will ignite and the composition of the most readily ignited mixtures of firedamp and air have been studied with a view to determine the liability of colliery equipment, such as flame and electric lamps, electrical apparatus generally, and explosives, to ignite firedamp. The ignition of mixtures of firedamp and air is dependent upon the composition, the temperature, and the time for which a sufficient volume of the gas is maintained at a suitable temperature. An intense source of heat, such as an electric spark, may cause an almost immediate ignition of the mixture, whilst owing to the lag on ignition, a similar firedamp–air mixture heated to its ignition temperature by a less intense heat may remain for 10 sec. before bursting into flame. For a given temperature, mixtures containing less methane exhibit, even at the highest temperatures, shorter lags on ignition than those containing more methane. A sustained flame will ignite all inflammable firedamp–air mixtures. Ignition by means of heated surfaces is largely dependent upon the extent of the surface, *e.g.*, a body possessing a large surface is more dangerous than a wire of the same temperature, and a wire is more dangerous than a frictional spark of equal temperature. In the case of electric sparks, capacity sparks are more dangerous than inductance sparks of equal energy. The character of the current supply, whether continuous or alternating, does not materially affect the incendivity of inductance sparks. The composition of the most easily ignitable mixture of methane and air depends upon the nature and source of the ignition, *e.g.*, for mixtures in closed heated vessels 5—7% CH_4, for mixtures ignited by sudden compression (such as might be produced in pockets on firing a shot) 6·5—8%, by heated wires 5—7%, by electric sparks 8—9%, by gas flames of short duration 8—10%, by the flame of some explosives 9% CH_4. A. W. HOTHERSALL.

Lag on ignition of firedamp. C. A. NAYLOR and R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 9, 1925. 16 pp.).—The relative ignition temperatures and the lag on ignition of methane–air mixtures and also of mixtures of air and ethane, propane, butane, and pentane respectively, have been determined. The incendivity of a heated surface depends upon its temperature, the duration of the time of contact with the gas, and the catalysing power of the surface which, by inducing flameless combustion, reduces the incendivity. Curves are given which show the variation of the observed ignition temperature with the size of the vessel and from these a judgment may be formed as to the ignition temperatures of the mixtures under adiabatic conditions. The lag on ignition is of shorter duration the higher is the temperature of the source of heat above the ignition temperature of the mixture, *e.g.*, with a methane–air mixture containing 6·5% CH_4 the lag was nearly 11 sec. at 700°, whilst it was only 0·01 sec. at 1175°. Mixtures of methane and air which contain an excess of oxygen have the lowest ignition temperature and the shortest lag on ignition and are therefore the most readily ignited. In the case of the other paraffin hydrocarbons studied, the most readily ignitable mixtures are those containing an excess of combustible gas. The order of relative ignitability of the paraffin hydrocarbons is as follows, methane–air mixtures being the most difficult to ignite: methane, ethane, propane, butane, pentane. For example, a lower-limit mixture of methane and air had a lag on ignition at 700° of 9·8 sec. compared with less than 0·5 sec. for a lower-limit mixture of ethane and air under similar conditions. A. W. HOTHERSALL.

Firedamp explosions within closed vessels. Effects of turbulence. G. B. MAXWELL and R. V. WHEELER (Safety in Mines Res. Bd. Paper No. 10, 1925. 12 pp.).—The effect of turbulence on the development of firedamp explosions in closed vessels has been studied in relation to the maximum pressure produced and the time taken to attain the maximum pressure, in order to determine the extent to which such turbulence modifies the character of an explosion which might occur within the casings of mining electrical machinery. The experiments were made in a vessel of 4 litres capacity fitted with a recording pressure gauge, the turbulence being induced by a small fan. Considerably enhanced pressures can be obtained from weak mixtures of methane and air (*e.g.*, 6% CH_4) when they are turbulent, but the most explosive mixtures, which contain 9—10% CH_4, are not much affected even by extreme turbulence. The maximum pressure obtainable from any quiescent mixture initially at atmospheric pressure when exploded in a closed vessel is about 105—110 lb. per sq. in., and the effect of turbulence is to increase this maximum pressure by about 4%. With both weak and strong mixtures, the effect of turbulence is greatly to increase the rapidity with which maximum pressure is attained, *e.g.*, with a mixture containing 9·65% CH_4 the times between ignition and attainment of maximum pressure were 0·069 sec. and 0·027 sec. for the quiescent and the turbulent mixtures respectively. Experiments were carried out in a 16·5-litre vessel formed from two flanged hemispheres with an adjustable gap between the flanges, the vessel being surrounded in some cases by an 8·5% methane–air mixture. In no case did ignition of the external mixture occur. The influence of turbulence in increasing the pressure and the rapidity with which it develops does not therefore affect the safety of "flange protection" devices for flame-proof mining electrical apparatus.
A. W. HOTHERSALL.

Explosive mixtures. VII. Influence of ethylene on the explosion limits of detonating [oxyhydrogen] gas. W. P. JORISSEN and B. L. ONGKIEHONG (Rec. trav. chim., 1926, 45, 162—165; cf. B., 1925, 980).—For mixtures of hydrogen and oxygen the explosion limits are determined as 6·1% and 92·3% H_2. For mixtures of ethylene

and oxygen the limits are 4·1% and 60·2% C_2H_4. The explosion limits for ternary mixtures have been determined and are represented on a triangular diagram. Except for mixtures consisting almost entirely of oxygen, the addition of ethylene tends to restrict the explosion zones of oxygen–hydrogen mixtures. W. Hume-Rothery.

Carbonisation research in Australia with a continuous vertical retort. R. E. Thwaites and J. Packer (J.S.C.I., 1926, **44**, 519—525 T).—A retort designed by Thwaites, the lay-out of auxiliary plant, their operation, and tests on them are described in detail. The retort is based on a small-scale plant experimented with in 1918 and embodies features of both Glover–West and Woodall–Duckham patterns. Means are provided for returning purified make-gas to the base of the retort with a view to speed up carbonisation and increase throughput by effecting a more rapid distribution of the heat, which was graded, being more intense at the bottom of the retort. This resulted in an increased throughput and a greater tar yield, but, as a strongly caking coal was used, also resulted in sticking, which was obviated by periodic poking. Owing to the relatively high outlet temperature and greater quantity of exit gases tar fog caused trouble, but this might be avoided by electrical precipitation. The coke was of good quality, partaking of the nature of semi-coke. Internal heating, it is concluded, is more suitable to a less strongly caking coal or blend of coals and to a wider retort. As a gas-making unit, the retort compares favourably with established types. R. A. A. Taylor.

Influence of the chemical and physical properties of coke on its combustibility. C. Holthaus (Stahl u. Eisen, 1926, **46**, 33—39).—The composition, silica content of the ash, apparent and true specific gravity, porosity, resistance to compression, friability, ignition temperature, gas content, and combustibility (or reactivity) of four different cokes have been determined. For most of these properties determinations have been made on samples of the same coke coming from different parts of the coke oven, viz., adjacent to the wall, the centre, and midway between wall and centre. In very few cases can definite relationships be observed between the various properties of the cokes. Simmersbach's assumption that the strength of the coke decreases with increasing silica content is not confirmed. All four cokes possessed nearly the same porosity as calculated from the apparent and true specific gravities. Determinations of porosity by microscopical observation of thin layers by transmitted light did not give satisfactory results. Useful qualitative information on the coke structure, however, was given by microscopical observation with reflected light. The ignition temperatures were determined by Bunte and Kölmel's method (B., 1923, 132 A), which gave very concordant results. The values and even the relative order of the ignition temperatures depended on particle size. Further heating of the coke in absence of air raised the ignition temperature. The combustibility (or reactivity) was determined by drawing a current of air at a constant rate over the coke, the temperature of which was maintained at 850°, and determining the percentage of the carbon dioxide formed which is converted into carbon monoxide. A. B. Manning.

Determination of apparent specific gravity of coke. M. Dolch (Z. angew. Chem., 1925, **38**, 889—891).—The apparent specific gravity of semi-coke, coke, or the like can be determined with an accuracy of about 2% by replacing the liquid used in the pyknometer by sand, shot, flaxseed, or gravel. A suitable pyknometer is described. T. S. Wheeler.

Properties of activated charcoal. P. Honig (Chem. Weekblad, 1926, **23**, 66—68; cf. Nellensteyn, B., 1925, 33; 1926, 35).—The differences in properties of various active charcoals which have been ascribed to differences in molecular structure are considered to be due to physical conditions, presence of impurities, porosity, etc., and the existence of "aliphatic" and "aromatic" carbons is denied. S. I. Levy.

Distillation analysis of petroleum, with special reference to Southern California crude oils. H. G. Vesper (Ind. Eng. Chem., 1926, **18**, 64—67).—The basis of the distillation is the four-point method of gasoline determination. A double distillation is made, the first distillation being carried to 260° (vapour). A condenser consisting of brass tubing in an ice bath is used, and for crude oils containing a high proportion of low-boiling hydrocarbons a supplementary condenser maintained at −73·3° by a mixture of alcohol or acetone and solid carbon dioxide is provided. The topping is carried out in a seamless copper flask of 4000 c.c. capacity at a rate not above 10 c.c. per min., but in the case of oils containing more than 10% of water and sediment a pressure still working at 100 lb. per sq. in. is used. 300—400 c.c. of the product of the first distillation are distilled in a 500-c.c. Hempel flask with a 15-cm. jack chain tower. The distillation curves of the naphtha and the A.S.T.M. distillation of gasoline are identical at the 50% and 90% points, but show divergence at the 20% point and the end point. The percentages over at 93·3°, 105°, 140°, 200°, and 215·5° are observed. The percentage over at 105° is multiplied by 5, 5·5, or 4 according as the percentage over at 93·3° is 5% or less, 5·5—6%, or 6·5% or more. The percentage over at 140° is divided by 0·9, and that at 215·5° by 1, and distillation cut at the lowest of these yields. For determination of lubricating oils and asphalt the residue from the first distillation is distilled at 4 cm. absolute pressure. 4% cuts starting at 232·2° are mixed to give a viscosity of 500 sec. Saybolt at 37·7°, this being the average viscosity of medium heavy lubricating stock. H. Moore.

Latent heats of vaporisation of distillates from paraffin-base petroleum. E. H. Leslie, J. C.

GENIESSE, T. W. LEGATSKI, and L. H. JAGROWSKI (Ind. Eng. Chem., 1926, **18**, 45—46).—A series of fractions of narrow boiling range were prepared from a high-grade paraffin-base oil. Each fraction was placed in a 500-c.c. vacuum bottle, and an electric current of 55 volts was passed by copper leads to a resistance coil of "chromel A" wire immersed in the liquid. A thermometer was inserted in the vapour space 0·5 in. below the cork. The vapour outlet tube passed to a Liebig's condenser, and no opportunity was afforded for reflux condensation. Heat to raise the temperature of the liquid and loss amounted only to 5—15% of the energy supplied, except at temperatures above 250°. The direct heat loss was determined by distilling substances of known latent heats. The latent heats observed were, for fractions boiling at 66·5°, 142·4 B.Th.U. per lb.; at 148·8°, 117·4 B.Th.U.; at 232·2°, 92·2 B.Th.U., and at 315·5°, 67·1 B.Th.U. The determined latent heats at the higher temperatures fall well below the values calculated by Trouton's rule.
H. MOORE.

Specific heats, heats of vaporisation, and critical temperatures of California petroleum oils. E. H. ZEITFUCHS (Ind. Eng. Chem., 1925, **18**, 79—82).—To ascertain the specific heat a known weight of oil was sealed up in a light steel cylindrical capsule of about 15 c.c. capacity. This was heated to the desired temperature in an electric tube furnace, and dropped into a Dewar flask containing 850 g. of water, the rise in temperature of the water being noted. The temperature of the oil in the capsule was measured by an iron-constantan thermocouple. The Dewar flask was calibrated by measuring the electric energy required to raise its temperature a given amount. A correction value for the empty capsule was determined by dropping it into the Dewar flask in the same manner as when oil was present. Tables are given, showing the specific heats of different products at various temperatures. To observe the critical temperature, oils were sealed up in glass capillary tubes of about 2 mm. bore and 8 mm. outside diam., and these were heated electrically until the meniscus disappeared. The critical temperature was found to depend on the vapour space above the oil. The heat of vaporisation at the mean boiling point of the oil was determined by Trouton's rule. Distillation of a number of samples showed that the temperature of the liquid was 3° to 20° above that of the vapour. H. MOORE.

Equilibrium boiling points [of motor fuels]. W. A. WHATMOUGH (Ind. Eng. Chem., 1926, 18, 43—45).—The apparatus used was that of Ormandy and Craven (B., 1924, 6), except that a pressure-equalising tube was fitted between the top of the boiler and the float chamber. When fuel was fed at the same rate as vaporised, constant boiling points were obtained for each fuel. To observe the equilibrium b.p. the fuel is distilled at the rate of about 30 c.c. per min. till the b.p. is steady, and then further observations are made at constant rates of 40 c.c. and 20 c.c. per min. The variation in b.p. should not be more than ±1°. The fuel assumes a spheroidal condition if the rate is more than 45 c.c. per min. Successful determinations were made with ¼ to 1 litre of fuel. The equilibrium b.p. tends towards the 85% point in paraffinoid distillates, but the 70—80% point is more usual with British motor fuels, and mixed fuels containing alcohol show a wider divergence. Alcohol markedly depresses the equilibrium b.p. and permits low induction pipe temperatures when used with kerosene.
H. MOORE.

Determination of lead tetraethyl in motor fuels. G. FERRERI (Giorn. Chim. Ind. Appl., 1925, 7, 625—626).—50 c.c. of the petrol are gently heated under a hood with 50 c.c. of concentrated hydrochloric acid (*d* 1·19) in a round-bottom flask of about 250 c.c. capacity fitted with a reflux condenser. In passing through the petrol the gaseous hydrogen chloride generated causes the precipitation of lead chloride, the turbidity at first increasing in intensity and afterwards disappearing. When the liquid has become quite clear again, the petrol and a considerable proportion of the aqueous liquid are expelled by distillation, the residue being transferred to a beaker and washed with boiling water. The liquid is then concentrated somewhat and mixed with absolute alcohol, the lead chloride being subsequently collected and weighed as usual. More accurate results are obtained by weighing as lead sulphate. For various petrols and petrol–benzol mixtures containing 0·421% of lead tetraethyl by weight the latter method gave from 0·413 to 0·425%.
T. H. POPE.

Neutralising action of activated decolorising earths on acidified lubricating oils. O. ECKART (Z. angew. Chem., 1925, 38, 885—887).—Chemically-activated decolorising earths, notwithstanding their slightly acid reaction, remove by adsorption the greater portion of the acid from acid lubricating oils in 45 min. at 95°. In this respect they are almost as effective as floridin or fuller's earth, which have an alkaline reaction due to the presence of carbonates. Dry earth is more active than damp material. The bleaching value of the activated earth is related to the neutralising power. T. S. WHEELER.

Coke macrostructure. H. J. ROSE (Fuel, 1926, 5, 57—64).—See B., 1925, 834.

Purifying used oils with fuller's earth. VON DER HEYDEN and TYPKE.—See XI.

Effect of ozone on oils. DOVER and APPLEBY. —See XII.

PATENTS.

Manufacture of fuel briquettes. Manufacture of combustible briquettes. P. E. WELTON (E.P. 244,623—4, 26.3.25).—(A) A cylindrical tubular briquette of granular structure is made by compressing a mixture of coal culm, by-product sulphite liquor, and a petroleum product of high m.p., and subsequently drying in an oven. The sulphite liquor serves as a binder, and the petroleum product as a waterproofing agent. The briquettes are broken, not

cut, into suitable lengths, the jagged ends so produced giving ease of ignition. The core of the briquette may be filled with some highly-combustible material, *e.g.*, a mixture of powdered coal, sawdust, and petroleum residue. (B) A briquette is made by mixing dry coal culm (about 1/16 in. size), granulated petroleum wax of high m.p., and the powdered residue obtained by evaporating sulphite waste liquor to dryness. Water is added to the thoroughly mixed material until it attains a putty-like consistency. It is then formed into tubular briquettes, and dried to dehydrate the binder, and to melt the petroleum wax, which serves as a secondary binder on cooling. Suitable proportions are: 100 pts. of coal culm, 2 of dried sulphite liquor, 3 of oil refinery residue wax, and 10 of water. Such a mixture gives good results if dried for 15 min. in an oven at 135°.

A. B. MANNING.

Manufacture of fuel briquettes. P. E. WELTON (E.P. 244,971, 26.3.25; cf. preceding abstract).—Fuel briquettes are made by mixing coal culm with a cellulose binder, prepared from sulphite-cellulose waste liquor, and a second binder consisting of a granulated petroleum solid of high m.p., which serves also as a waterproofing material. The mixture is extruded through a die and broken into briquette lengths, and the briquettes are dried and then heated to a temperature sufficiently high to melt the waterproofing material. The culm is dried, and graded to consist of equal proportions of 16-, 48-, 75-, and 100-mesh size before mixing, all the operations being performed by a continuously acting machine.

A. B. MANNING.

Manufacture of coal briquettes. L. H. F. GUILLEMOT (F.P. 594,080, 1.9.24).—Powdered coal is mixed with 20% of a viscose solution, pressed, and heated to 90—100°. The viscose coagulates and a fuel of great strength and uniformity is obtained.

A. GEAKE.

Binding material [for briquettes]. BRIKETT-HARZ-GES. M.B.H. (G.P. 418,991, 3.4.25; Addn. to 393,546; cf. E.P. 210,548, also G.P. 405,182, B., 1925, 89, 196).—Acid is first removed from acid resin with alkali; the resin then retains sufficient alkali for the subsequent treatment. A. GEAKE.

Detection of small amounts of firedamp with an electric mine-lamp. GES. FÜR PRAKTISCHE GEOPHYSIK M.B.H. (G.P. 398,960, 5.1.23).—An electric mine-lamp is provided with a container holding finely-divided adsorbent charcoal which adsorbs a relatively greater amount of methane than of air from the atmosphere of the mine. After removing the lamp from the mine the adsorbed gases are expelled from the charcoal by heating it by means of an electric wire resistor and may be tested for methane by any suitable means, *e.g.*, by explosion.

A. R. POWELL.

[Production of] water-gas. J. C. SMITH (Can. P. 245,557, 27.5.22).—Steam is brought into contact with charcoal heated to such a temperature that the latter combines with the oxygen of the water to form carbon monoxide with the production of heat, which is absorbed by the decomposition of the water. An electric current is passed through the charcoal. A. B. MANNING.

Still [for coking petroleum]. P. YOUNG, Assr. to TEXAS CO. (U.S.P. 1,564,312, 8.12.25. Appl., 5.6.20).—The bottom of the still is formed of thick plates bound together by thin plates. The latter adapt themselves to the changes caused by thermal expansion and can readily be replaced as required. T. S. WHEELER.

Calcining material [petroleum coke]. A. S. WALDEN, Assr. to NAT. CARBON CO. (U.S.P. 1,564,730, 8.12.25. Appl., 24.3.21).—Petroleum coke is introduced into the upper end of an inclined rotary kiln and is preheated by the gases coming from the lower portion, into which air is admitted to burn part of the combustible gases evolved by the coke. A reducing atmosphere is thus maintained so that none of the hot coke is oxidised. Any residual combustible gas is consumed by admitting a small quantity of air to the upper portion of the kiln, where the coke is not hot enough to be attacked.

T. S. WHEELER.

Dry-distillation apparatus [for calcium acetate]. N. STATHAM, Assr. to WEST VIRGINIA PULP AND PAPER CO. (U.S.P. 1,566,778, 22.12.25. Appl., 12.12.21).—An apparatus for distilling calcium acetate comprises a rotating kiln divided into a preliminary drying chamber and three other chambers, of which the first is maintained at about 350°, the second at 450°, and the third at 550°. Partitions between the chambers, perforated to permit the heated material to pass, minimise gas convection currents between one chamber and another. A good yield of acetone is obtained. T. S. WHEELER.

Carbonisation of wood. R. A. H. P. CHAPELLE (F.P. 592,812, 7.4.24).—Wood is carbonised by treatment with hot gases containing a high percentage of carbon monoxide. L. A. COLES.

Autothermic distillation of solid fuel. J. É. HAENNIG (F.P. 594,186, 26.2.25).—The residue is automatically withdrawn from an oven fed continuously with coal or the like. The lower part of the oven is cooled by gas circulated at a controlled speed, and heat thereby exchanged between the red-hot coke and the charge. A. GEAKE.

Partial conversion of coal into light hydrocarbons. COMP. DES MINES DE VICOIGNE, NOEUX ET DROCOURT (F.P. 591,958, 11.3.24).—Finely-pulverised coal is conveyed, suspended in a current of gas, *e.g.*, steam, hydrogen, or coal gas, through a distilling apparatus heated to a suitable temperature.

A. B. MANNING.

Separating condensable [hydrocarbon] vapours from gases. J. C. SWAN (U.S.P. 1,563,028, 29.12.25. Appl., 15.12.24).—The gas from which condensable hydrocarbons are to be removed is passed through a column of liquid maintained below

the condensing temperature of the desired hydrocarbons by means of a refrigerating liquid in contact with external and internal cooling surfaces of the column. B. M. Venables.

Purifying gases obtained from coal or other fuels. A. Sander (G.P. 418,109, 30.12.23).—Gases containing ammonia and carbon dioxide are freed from tar and passed through a solution containing at least 20% of calcium chloride. Calcium carbonate is filtered off, and ammonium chloride obtained from the filtrate by evaporation. The apparatus required is small and simple, and the use of sulphuric acid is avoided. A. Geake.

Fuels [for internal-combustion engines]. A. E. White. From General Motors Corp. (E.P. 245,281, 20.1.25).—A fuel which has a relatively high critical compression pressure and which lubricates, and prevents the formation of a deposit on, the valve stems and other engine parts, consists of the usual fuel, such as gasoline or kerosene, mixed with a knock-reducing compound (*e.g.*, lead tetraethyl), a halide (ethylene or propylene dibromide, or both), which produces during the combustion compounds which do not form deposits, and a lubricant which is stable at high temperatures, such as a chlorinated or brominated aromatic oil, *e.g.*, monochloronaphthalene. For example, to a gallon of gasoline or kerosene there may be added 4 c.c. of a mixture of 40 pts. of lead tetraethyl, 18 pts. of ethylene dibromide, 7 pts. of monochloronaphthalene, and 5 pts. of propylene dibromide. A. W. Hothersall.

Alcohol fuel. F. Lioud (F.P. 593,606, 9.12.24).—A fuel is prepared by bubbling alcohol vapour through crude benzol. S. Binning.

Cracking and separating of mineral oils. V. L. Oil Processes, Ltd., and O. D. Lucas (E.P. 244,875, 31.10.24).—Oil is preheated to about 400° and sprayed into a preliminary cracking retort heated to about 500° and containing a layer of coke or other carbonaceous material kept in motion by a conveyor. The oil is cracked in the vapour phase, carbon is deposited on the coke, and any excess is periodically removed. The vapours pass to a second retort provided with a catalyst, and also heated to about 500°. The vapours from the second retort pass to a series of condensers, in the first of which the heavier oils are condensed, and in the succeeding ones the medium and light constituents. The medium constituents are preheated and returned to the spraying device of the first retort, the light products are collected, and the gases led to a gasholder. A portion of the permanent gases may be returned to the first retort to assist in carrying over the vapours, and a small amount of ammonia may be mixed with them to supply nascent hydrogen and assist in the production of saturated compounds. In the second chamber cracking is effected without substantial deposition of carbon. The cracking apparatus may be connected directly to an installation for the low-temperature distillation of shale, coal, or lignite. H. Moore.

Distilling petroleum oils. E. M. Clark, Assr. to Standard Development Co. (U.S.P. 1,566,828, 22.12.25. Appl., 24.4.20).—The heat retained by the residual oil and by the still structure at the end of a pressure distillation of a heavy oil can be partially utilised by withdrawing the still fire and passing steam through the oil in the still, when about 60% can be distilled and refined. T. S. Wheeler.

Apparatus for treating [cracking] hydrocarbons. F. C. Van de Water and F. R. Sunderman, Assrs. to Petroleum Laboratories, Inc. (U.S.P. 1,567,212, 29.12.25. Appl., 17.5.23).—The vapours from a still pass into a cracking tube underneath the still, the tube being enclosed in a protective casing. The casing and the still are heated from a common source of heat, and cool air is circulated through the casing around the cracking tube. H. Moore.

Series separation of crude petroleum. A. R. Earl and T. W. Reeves (U.S.P. 1,567,429, 29.12.25. Appl., 25.8.23).—Mineral oil is continuously distilled by heating it under pressure by means of steam, releasing the pressure, and passing the oil into an expansion zone where it partially vaporises. The vapours are fractionally condensed, and the unvaporised oil is withdrawn and further heated, without permitting vaporisation, and passed into an expansion zone at a pressure below atmospheric, where the lighter products vaporise. These are condensed and the unvaporised oil is withdrawn and further heated with superheated steam under pressure to prevent vaporisation. The heated oil is again passed into an expansion zone at a pressure below atmospheric, the vapours evolved are condensed, and the unvaporised oil is withdrawn. H. Moore.

Evaporating still [for mineral oil]. D. L. Newton (U.S.P. 1,567,455, 29.12.25. Appl., 7.8.22).—A vertical casing is divided by horizontal partitions into four compartments; to the first or lowest compartment the oil or liquid to be distilled is supplied and this compartment communicates through a number of tubes of small bore with the fourth compartment or vaporising chamber from which vapours are drawn off. A number of tubes of large bore lead downwards from the fourth compartment to the second, which contains residual oil, into which steam is injected. A heating medium is supplied to the third compartment which surrounds both sets of tubes. B. M. Venables.

Converting [cracking] hydrocarbon oils. J. H. Adams (U.S.P. 1,568,016, 29.12.25. Appl., 12.10.22).—The oil is subjected under pressure to direct contact with an electric resistance, immersed in the oil and heated to cracking temperature. H. Moore.

Treatment of hydrocarbons. C. N. Forrest and H. P. Hayden, Assrs. to Barber Asphalt Co. (U.S.P. 1,568,018, 29.12.25. Appl., 15.6.20).—Inert refractory material is saturated with hydrocarbon

material to be destructively distilled and gasified, and is passed progressively through a sphere of reaction, meeting, towards the exit end, a limited counterflow of combustion agent. Coke produced by cracking of the hydrocarbon is thereby consumed, and the carrier material is restored to its original clean condition. H. MOORE.

Obtaining commerical gasoline from natural-gas gasoline. H. J. MACKENZIE and A. W. STEENBERGH, Assrs. to DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (U.S.P. 1,569,433, 12.1.26. Appl., 26.9.23).—Gasoline condensed from natural gas by compressing it and subsequently cooling it to normal temperature, is removed and blended with other gasoline, pressure being maintained throughout the process. After cooling the mixture to a low temperature under pressure, dissolved gas is removed by releasing the pressure and subsequently bringing the liquid gradually to atmospheric temperature. L. A. COLES.

Heat treatment [cracking] of hydrocarbons. H. R. BERRY, Assr. to DYNAMICS CORP. OF AMERICA (U.S.P. 1,569,532, 12.1.26. Appl., 6.12.24).—Hydrocarbon oil and steam at a suitable temperature are introduced continuously and separately into an enlarged zone at such rates that any carbon produced by cracking of the hydrocarbons is converted into carbon oxides by combination with oxygen derived from the steam, whereby deposition of carbon upon the wall of the chamber is prevented and the specific gravity of the resulting hydrocarbons is reduced. J. S. G. THOMAS.

Purification or separation of aliphatic hydrocarbons. E. JENA (G.P. 419,301, 31.3.22).—Crude, partly refined, or distilled hydrocarbons are purified by extraction with chlorohydrins or with ethyl phthalate, which are more effective than alcohol or organic acids. L. A. COLES.

Purification of hydrocarbons. J. SCHÜMMER (G.P. 419,302, 27.3.24).—Hydrocarbons are purified by passing them in the form of vapour over acid-reacting porous material with a large surface, precautions being taken to prevent deposition of solid resinous condensation products, or by passing them successively over acid-reacting and alkaline-reacting porous material. L. A. COLES.

Purifying [used lubricating] oils. BRITISH THOMSON-HOUSTON CO., LTD., Assees. of C. VAN BRUNT (E.P. 235,861, 28.5.25. Conv., 23.6.24).—A resinate of a heavy metal, preferably manganese, is added to used lubricating oil before clarifying it by a solution of water-glass. A 20% solution of the resinate in benzol or oil is preferably used in the proportion of 1 pt. of resinate to 200 pts. of the oil to be purified; 0·1% of stearic acid may be added to the resinate. After addition of the resinate 1 to 4 oz. of a solution of water-glass of 40° B. (d 1·384) is added per gallon of the oil. After mixing, the mixture is floated on water heated to 80°, when the impurities subside as sludge. Any benzol used is removed along with the volatile ingredients in subsequent rectification. None of the reagents used remains in the clarified oil. H. MOORE.

Purification of montan wax. SCHLICKUM-WERKE A.-G., and E. KOEH (G.P. 419,161, 29.1.24).—The wax is distilled with superheated steam and hydrogen. S. BINNING.

Inclined coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS CO. (U.S.P. 1,570,871, 26.1.26. Appl., 29.9.21).—See E.P. 218,690; B., 1924, 777.

Recovery of readily liquefiable constituents [gasoline] from hydrocarbon gas mixtures. E. C. R. MARKS. From CARBIDE AND CARBON CHEMICALS CORP. (E.P. 245,860, 18.10.24).—See U.S.P. 1,523,313; B., 1925, 163.

Apparatus for preventing the aeration of centrifugally treated liquids [lubricating oils]. AKTIEBOLAGET SEPARATOR, Assees. of S. H. HALL (E.P. 241,194, 6.10.25. Conv., 8.10.24).—See U.S.P. 1,561,784; B., 1926, 33.

Manufacture of incandescence gas mantles. T. TERRELL (E.P. 245,891, 21.11.24).

Filter (U.S.P. 1,567,989).—See I.

Cement and combustible gas (E.P. 228,129).—See IX.

Organic compounds (E.P. 244,830).—See XIII.

Insecticides (E.P. 245,179).—See XVI.

III.—TAR AND TAR PRODUCTS.

PATENTS.

Separation of low temperature tar into phenols and hydrocarbons. H. E. POTTS. From ZECHE M. STINNES (E.P. 245,633, 7.3.25).—A mixture of low-temperature tar with dilute alcohol is saturated with ammonia, cooled, and allowed to settle into two layers. Phenols and neutral oils are recovered from the lower and upper layer, respectively, by heating the layers, after separation, to drive off water, alcohol, and ammonia, which are used again in the process. For example, a mixture of 500 kg. of tar, b.p. 180—250°, and containing 32% of phenols, 2% of basic compounds, and 66% of neutral oil, with 534 kg. of 56% alcohol, is saturated with ammonia, about 84 kg. being required. After separation, the phenol layer is heated to 90—95°, and the neutral oil layer to 180°, 94% of the total phenols (purity 99%), and 99% of the neutral oils (containing 5% of phenols) being recovered. L. A. COLES.

Separation of oil mixtures. [Separating phenols from low-temperature tar.] A. IRINYI (G.P. 417,615, 25.6.24).—Low-temperature tar oil containing phenols, in vapour form, *e.g.*, the vapour coming from the still, is liquefied by compression at the boiling point and blown with an equal volume of water at 100° through opposing nozzles into a closed vessel. If the oil has b.p. 250°, the

temperature in the vessel will be maintained without addition of heat, at about 200°. The pressure in the vessel must be sufficient to maintain the water in the liquid state. Under these conditions the phenols present in the oils are rapidly extracted. The process may be varied by mixing the oil vapour and steam before compression. T. S. Wheeler.

Purification of hydrocarbons. (G.P. 419,302). —See II.

IV.—DYESTUFFS AND INTERMEDIATES.

See A., Feb., 158, **Sulphuryl chloride. New chlorinating agent. Preparation of polychloro-derivatives of toluene** (Silberrad). 162, **Products of action of sulphites on 1 : 8-dinitronaphthalene** (Bucherer and Barsch). 164, **Nitrogen tetroxide as reagent for diazotisation** (Houston and Johnson); **Sulphonation of *p*-chlorophenol** (Gauntlett and Smiles). 172, **Catalytic hydrogenation of anthraquinone, phenanthraquinone, and benzanthrone** (Von Braun and Bayer). 173, **Preparation of hydrogenated polynuclear quinones** (Skita). 174, **Reduction products of hydroxyanthraquinones** (Miller and Perkin); **Action of hydrazine hydrate on phenanthraquinone** (Dutt). 183, **Dye formed by action of ethyl oxalate on magnesium pyrryl bromide** (Godnev and Naryschkin).

Patents.

Brown trisazo dyestuffs [for leather]. B. Vossen, Assr. to Grasselli Dyestuff Corp. (U.S.P. 1,565,344, 15.12.25. Appl., 17.12.24).—An amine is diazotised and coupled with resorcinol or a derivative thereof, and the product is coupled successively with a diazotised aminonaphthol and a diazotised amine, to yield a dyestuff giving fast even brown shades on leather. For example diazotised sulphanilic acid is coupled with resorcinol and the product coupled first with diazotised 1-amino-8-naphthol-4-sulphonic acid and then with diazotised *p*-nitroaniline. A dyestuff giving yellow-brown shades on leather is obtained. T. S. Wheeler.

Manufacture of 2-aminoanthraquinone. O. D. Cunningham, Assr. to Nat. Aniline and Chemical Co. (U.S.P. 1,564,210, 8.12.25. Appl., 7.6.21).—60 pts. of sodium anthraquinone-2-sulphonate, 47·5 pts. of crystallised barium chloride, 45 pts. of ammonium chloride, and 1100 pts. of 23% aqueous ammonia are heated in an autoclave with agitation at 185° for 20 hrs. 2-Aminoanthraquinone is formed in 82% yield. T. S. Wheeler.

Production of *m*-amino-*p*-tolyl methyl ether. C. G. Derick, T. H. Leaming, and W. M. Ralph, Assrs. to Nat. Aniline and Chemical Co. (U.S.P. 1,564,214, 8.12.25. Appl., 9.3.20).—*m*-Nitro-*p*-cresol is methylated with methyl sulphate in presence of an excess of caustic soda at 45—100°, and the resulting mixture while still hot is treated with sodium sulphide solution and steam-distilled to recover the *m*-amino-*p*-tolyl methyl ether formed. T. S. Wheeler.

Controlling chemical reactions. [Preparation of indoxyl.] H. H. Dow, Assr. to Dow Chemical Co. (U.S.P. 1,564,218, 8.12.25. Appl., 8.12.20).—Phenylglycine mixed with a neutral hydrocarbon oil, such as kerosene, is added to molten caustic soda. The oil used has a boiling point between the temperature of the molten caustic soda and the decomposition temperature of phenylglycine, and its vaporisation maintains the temperature of the melt below the decomposition point of phenylglycine, while the vapour evolved protects the melt from the action of air. T. S. Wheeler.

4-Nitro-1-acetnaphthalide-6 (or 7)-monosulphonic acid. W. M. Ralph, Assr. to Nat. Aniline and Chemical Co. (U.S.P. 1,566,425, 22.12.25. Appl., 29.11.20).—Cleve's acid (1-naphthylamine-6(7)-sulphonic acid) is acetylated by treatment with acetic anhydride and sodium acetate and the product is dissolved in sulphuric acid and nitrated with a mixture of nitric and sulphuric acids. The mixture is then added, with cooling, to sodium carbonate solution when the sodium salt of 4-nitro-1-acetnaphthylamide-6(7)-sulphonic acid is formed and salted out by the sodium sulphate also produced. T. S. Wheeler.

Preparation and separation of 1-aminonaphthalene-8- and -5-sulphonic acids. G. Poma (F.P. 560,318, 23.12.22. Conv., 24.12.21.).—Mixtures of 1-nitronaphthalene-8- and -5-sulphonic acids, obtained by nitrating naphthalene-1-sulphonic acid in sulphuric acid solution by the addition of sodium nitrate followed by dilution with water, precipitation by the addition of salt, and filtration, are dissolved in water, neutralised with sodium hydroxide or carbonate, or with magnesium carbonate, and reduced by treatment at 50° with hydrogen under 4—5 atm. pressure, or with sulphur dioxide, in the presence of finely-divided catalysts, such as nickel, cobalt, copper, or iron, or their oxides. After filtration, 1-aminonaphthalene-5-sulphonic acid is precipitated by acidification of the solution, and the insoluble sodium 1-aminonaphthalene-8-sulphonate is separated from the catalyst after conversion into a soluble *N*-aryl derivative, *e.g.*, by treatment with aniline or *p*-toluidine. L. A. Coles.

Preparation of the anilide of 2-naphthol-3-carboxylic acid. Chem. Fabr. Rohner A.-G. Pratteln (Swiss P. 108,072, 19.3.24).—The anilide is obtained by heating a solution in a tertiary base, *e.g.*, dimethylaniline, of equimolecular proportions of aniline and 2-naphthol-3-carboxylic acid, in the presence of phosphorous trichloride. L. A. Coles.

Preparation of secondary bases of the naphthalene series [dinaphthomethylamines]. H. Rupe (Swiss P. 108,704 and 109,066, 30.6.23).—When α- and β-naphthonitriles are reduced with hydrogen in the presence of nickel they yield respectively αα- and ββ-dinaphthomethylamine, which are of use in the production of dyestuffs and drugs (cf. Rupe and Becherer, A., 1923, i., 1202). L. A. Coles.

Preparation of $O:O$-diacetyldiphenolisatin. F. Hoffmann-La Roche u. Co. A.-G. (Swiss P. 108,872, 9.4.24. Addn. to 100,806; cf. E.P. 221,976, B., 1925, 26).—The compound is obtained by acetylating diphenolisatin with acetic anhydride, with the addition of a diluent, if necessary, at temperatures above 100°, *e.g.*, at 120—130°. L. A. Coles.

Feeding solids and semi-solids to chemical apparatus (U.S.P. 1,565,249).—See I.

Material for combating plant pests (G.P. 419,461).—See XVI.

Amines and substitution products (U.S.P. 1,564,631).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Specific gravity of wool and its relation to swelling and sorption in water and other liquids. A. T. King (J. Text. Inst., 1926, 17, T. 53—67).—The apparent density of alcohol-extracted wool is approximately constant for all but the coarsest, kempy kinds. The following values for the apparent sp. gr. of dry wool at 25° referred to water at 25° were obtained in the media named :—Methyl alcohol 1·408, water 1·3965, ethyl alcohol 1·388, glycerol 1·334, light naphtha 1·328, paraffin B.P. 1·320, amyl alcohol 1·318, carbon tetrachloride 1·309, oleic acid 1·308, nitrobenzene 1·306, olive oil (dry) 1·306, toluene 1·306, benzene 1·304. The apparent density in water of wool conditioned in an atmosphere of a definite relative humidity is connected with the percentage regain, R, according to the expression $D=(100+R)/(71{\cdot}6076+R)$; R can be determined from a density measurement with reasonable accuracy. The regain is substantially the same when wool is conditioned in saturated air ("saturation regain") or placed direct into liquid water ("soaking regain"). The high values for apparent densities in methyl and ethyl alcohols suggest that these behave like water towards wool. This is confirmed by the observation that wool also absorbs their vapours. The "saturation regains" at 25° in water, methyl alcohol, ethyl alcohol, and benzene vapours are approximately 33, 26·3, 21·3, and less than 0·5%, respectively. High values of the apparent density are due to sorption of the medium and swelling, and the minimum values in liquids like benzene may be taken as the true density. Using this value (or rather, the specific volume) it is possible to calculate from the apparent specific volume in a given medium, the known specific volume of that medium, and the saturation regain, the amount of swelling which occurs when wool is saturated with the medium. The value for water is 36·3%, agreeing with direct measurements on single fibres, and the value for ethyl alcohol is 28·5%, by volume. There appears to be some compression of the sorbed medium which can be expressed as a "compression factor," the volume of medium which causes a swelling of 1 c.c.; the figure for water is 1·18, and for ethyl alcohol 1·22. Figures are also recorded for the density of wool containing moisture represented by 0—33% regain in benzene and in water at 25°, and also for the compression factor for the water sorbed and the swelling over this range. It is also shown how specific gravity figures can be used to calculate the regain of a sample of wool, the percentage of wool in a scoured sample, and the proportion of cotton in a cotton and wool mixture. The work described also throws some light on the penetration of wool by solvents and oils, the setting of wool yarns by alcohol, and the effects of salt on the resilience and dyeing and milling properties of the fibre. These are briefly discussed.

J. C. Withers.

Moisture relations of cotton. Absorption of water by cottons of various origins. A. R. Urquhart and A. M. Williams (J. Text. Inst., 1926, 17, T. 38—45).—Variations between the moisture contents of different growths of cotton are insufficient to use for the purposes of identification. They are mainly due to the moisture capacity of the impurities, for after washing with hot water all the cottons tested had almost exactly the same absorptive powers. Further purification by scouring and bleaching lowers the hygroscopicity progressively, and dyeing with basic dyes also causes a reduction which cannot wholly be explained by the loading of the cotton with non-hygroscopic material.

J. C. Withers.

Chemical analysis of cotton. Ash content and ash alkalinity of typical cottons. R. G. Fargher and M. E. Probert (J. Text. Inst., 1926, 17, T. 46—52).—"Ash alkalinity" is defined as the number of c.c. of N-acid required to neutralise the ash from 100 g. of cotton. The table summarises data obtained from hand-combed samples :—

Cotton.	Ash %.	Ash alkalinity.	Alkalinity per gram of ash.
N. American (12 samples)	1·17	16·46	14·1
S. American (7)	1·16	16·67	14·3
Indian-American (7)	1·25	18·6	14·9
Other outside growths of American (15)	1·47	22·5	15·3
Egyptian (home growths) (6)	1·20	18·28	15·28
Egyptian (outside growths) (4)	1·26	20·04	15·86
Native Indian (10)	1·28	19·2	15·1
Sea Island (6)	0·98	14·9	15·2

The results show that the ash content of cotton which is free from adhering sand and dirt is not as high as is often quoted. The least variable figure within a group is the alkalinity per gram of ash, and its determination is sufficiently exact to serve as a distinguishing test between American and Egyptian growths. Further, because of its constancy, experiments involving changes in the ash content of cotton (such as tests on scouring) can be based on the simple titration of the ash without weighing it.

J. C. Withers.

Mildew in cotton goods. Growth of mould fungi on sizing and finishing materials. L. E. Morris (J. Text. Inst., 1926, 17, T. 1—22).—Mainly botanical. The botanical and chemical characteristics of the flours, starches, dextrins, and gums commonly used in sizing are reviewed, and

as a result of the experiments they are grouped according to their liability to become mildewed in the form of pastes. Tapioca flour, sago, and farina are least liable and farina treated with caustic soda is almost immune. J. C. Withers.

Mildew in cotton goods. Growth of mould fungi on steeped wheat flour. L. E. Morris (J. Text. Inst., 1926, 17, T. 23—37).—When wheat flours are used for sizing, they are usually "steeped" for a period, during which, under the influence of atmospheric infection, a certain amount of fermentation proceeds. The opinion is held that steeping lessens the liability of mildew to develop subsequently on the sized cloth. The chemistry of the process is discussed and experimental evidence is given to show that whilst there is some justification for the above opinion, there are considerable specific differences between the various mildew-producing fungi in their ability to grow on steeped flour. The process is effective, for example, against *Cladosporium* and *Fusarium* but not against *Aspergillus* and *Penicillium.* J. C. Withers.

Pectin content of flax fibre. W. Honneyman (J. Text. Inst., 1925, 16, T. 370—374).—Various retted flaxes and flax extracts have been tested by the method of Nanji, Paton, and Ling (J.S.C.I., 1925, 44, 253 T). It appears that the pectin content of the flax fibre bundle when separated from cortical tissues is not more than about 2%. This residue, however, is associated mainly with the middle lamella binding the ultimate fibres, and is the cause of the difficulty of bleaching flax satisfactorily. J. C. Withers.

Test to distinguish between viscose and cuprammonium artificial silks. O. S. Rhodes (J. Text. Inst., 1926, 17, T. 75—76).—The reagent used contains silver nitrate (1%), sodium thiosulphate (4%), and sodium hydroxide (4%), and is more effective than an ammoniacal solution. Due to its sulphur content, viscose is stained brown when left in the boiling solution for 1 min., whereas cuprammonium silk remains white unless it is one of the varieties which contains oxycellulose, when it may become grey or light brown. The stain on viscose is matched by a 2·5—4% dyeing with Chlorazol Brown M. Nitro-silks behave like viscose but may be distinguished by the diphenylamine test.
J. C. Withers.

Maturing of viscose, with special reference to colloid-chemical changes. R. Bernhardt (Kunstseide, 1925, 7, 169—174, 193—198; Chem. Zentr., 1926, I., 793).—Sodium dicellulose xanthate is formed by treating matured alkali-cellulose with about 10% of its weight of carbon disulphide at a suitable temperature. A portion of the excess carbon disulphide is converted into sodium carbonate and sodium sulphide by the sodium hydroxide, and a portion remains in suspension in the viscose. In the ageing process, the greater part of the xanthate polymerises to tetracellulose xanthate previous to the spontaneous gelatinisation. Carbon disulphide is generated continuously during maturing, part of it being converted simultaneously into hydrogen sulphide and carbon dioxide. The maturing of viscose is a colloid-chemical process, accompanied by definite chemical changes. Variations in the colloidal properties during the ageing process are established by determinations of the size of the particles, of the viscosity, and of the gelatinising properties, and chemical changes by analyses of the gaseous products, by the deepening of the colour, and by the increasing turbidity. L. A. Coles.

Change of alkalinity of liquor during cooking of soda- and sulphate-cellulose. C. Kullgren (Papier-Fabr., 1926, 24, 20—23).—The hydroxyl-ion concentration of the liquor in the boiling of fir wood was determined by Koellichen's method (A., 1900, ii, 395) in which the rate of decomposition of diacetone-alcohol is measured. When the initial liquor is $1·9N$-sodium hydroxide the concentration of hydroxyl ions falls fairly rapidly during the first $3\frac{1}{2}$ hrs.' boiling to about $^1/_7$ of its initial value. If part of the sodium hydroxide is replaced by sodium sulphide ($1·47N$-NaOH$+0·44N$-Na_2S) the concentration of hydroxyl ions is lower throughout the boil and it falls more rapidly. It is believed that this is the cause of the 10% higher yield of cellulose obtained under the latter conditions. A. Geake.

Alkali loss in sulphate-cellulose manufacture. R. Sieber (Papier-Fabr., 1926, 24, 6—7).—The principal losses of sulphate in the manufacture of sulphate-cellulose from wood are as flue dust (20—25 kg. per ton of pulp) and in the wash water from the pulp (60—70 kg. per ton). The total loss is at least 115—120 kg., and usually it is 140—170 kg., corresponding to 16—19%. A. Geake.

Determination of the strength of cellulose fibres. F. Rühlemann (Papier-Fabr., 1926, 24, 1—6).—To determine the strength of individual fibres from cellulose pulp for paper making, these are mounted across a gap in a piece of nitrocellulose film. After inserting in the apparatus, the supporting nitrocellulose is burnt through with an electrically-heated platinum needle. The gap is not more than 1·5 mm. long. The load is applied at a constant rate by the addition of water to a cup from a burette. Extension is measured by photographing with a micro-kinematograph and measuring an enlarged projection. A. Geake.

Time factor and yield value of cellulose esters. S. E. Sheppard, E. K. Carver, and S. S. Sweet (Ind. Eng. Chem., 1926, 18, 76—77; cf. B., 1925, 912).—Films of cellulose esters are not perfectly elastic and flow slightly even under small loads. The rate of flow is proportional to some high power of the load, instead of being proportional to the difference between the load and yield value, as is the case with many plastics. The yield point as obtained by the ordinary strength-testing machines depends largely upon the speed at which the machine is run. It is the point at which the flow is equal to the rate at which the pull is applied. A graph showing stress-strain diagrams with identical material but with

different rates of pull, proves that the yield point and breaking load are merely accidental values depending on the rate at which the pull is applied. The logarithm of the rate of pull is roughly proportional to the breaking load. D. WOODROFFE.

Colloidal properties of nitrocellulose sols in mixed solvents. A. HIGHFIELD (Trans. Faraday Soc., Jan., 1926. Advance proof).—Nitrocellulose contains both strongly polar (hydroxyl) groups and weakly polar (hydrocarbon) groups, and the best solvents for it contain these two classes of groups in suitable proportions. This accounts for the better solvent power of mixed than of pure solvents. The viscosity of a 4% sol was taken as a measure of the solvent power, and a considerable number of mixed solvents were investigated. The viscosity of the sol is also affected to some extent by the viscosities of the constituents of the solvent. The composition of the best solvent mixture of, for example, water, alcohol, and ether, depends on the nitrogen content of the nitrocellulose. As the nitrogen content decreases the proportion of polar (hydroxyl) groups increases, necessitating a corresponding increase in hydroxyl groups (water and alcohol) in the solvent. The viscosity is lowered by the addition of small quantities of hydrochloric acid and especially of sodium hydroxide. A. GEAKE.

Action of diazomethane on cellulose. NIERENSTEIN.—See A., Feb., 154.

Lignification. Nature of lignin and its determination in timbers. Polysaccharides and aromatic constituents of the cell wall. MEHTA.—See A., Feb., 209.

Disinfectants from chlorinated sulphite-cellulose waste lye. HILPERT.—See XXIII.

Cause of "gulf" disease. BIGINELLI.—See XXIII.

PATENTS.

Insecticide for moths. W. STRAUB (G.P. 419,464, 13.1.23).—An insecticide for removing moths from furs, woollen goods, and the like consists of a halogenated aromatic acylalkylamide, such as *N*-ethylacetamidotrichlorobenzene, either alone, in solution in a suitable solvent, or in admixture with a finely powdered basic substance, such as lime or magnesia, which does not decompose the amide. A. R. POWELL.

Converting vegetable fibre to pulverised form. E. B. PUTT (U.S.P. 1,565,864, 15.12.25. Appl., 15.7.25).—Sulphite wood pulp is boiled with 20 times its weight of 10% sulphuric acid for 30 min. The treated fibre is readily pulverised and serves as a base for ointments, dentifrices, and the like. T. S. WHEELER.

Production of cuprammonium cellulose solutions for spinning artificial silk. M. HÖLKEN (G.P. 420,422, 28.12.24).—Air and gas bubbles are expelled from constituents, particularly from the cellulose, for use in the preparation of cuprammonium cellulose solutions. For example, cellulose impregnated with cupric hydroxide is subjected to a high vacuum prior to solution. L. A. COLES.

Manufacture of non-deliquescent solid material from sulphite-cellulose waste liquor. A.-G. FÜR ANILIN-FABR. (F.P. 592,119, 24.1.25. Conv., 24.1 and 7.4.24).—Hygroscopic material present in sulphite-cellulose waste liquor is separated by dialysis, is decomposed in the liquor, or is rendered non-hygroscopic by neutralisation, or the ligninsulphonic acid is precipitated free from hygroscopic material. For example, waste liquor from which iron and calcium compounds have been removed is treated, at 100°, with sodium hydroxide solution, *d* 1·357. After some time, the liquor is neutralised with hydrochloric acid, and non-hygroscopic constituents are precipitated by the addition of salt, filtered off, and dried in a vacuum. Alternatively, the liquor is subjected to alcoholic fermentation and subsequently evaporated in a vacuum, or alcohol is added to it and the precipitated material is removed and dried, or it is heated with phenol, followed by the addition of hydrochloric acid and evaporation to dryness. L. A. COLES.

Manufacture of cellulose solutions. A. CLASSEN, Assr. to COMMERCIAL ALCOHOL CO., LTD. (U.S.P. 1,570,553, 19.1.26. Appl., 18.8.24).—See E.P. 236,281; B., 1925, 707.

White liquid paste for paper and cardboard. L. G. F A. WATTECAMPS (E.P. 246,381, 3.7.25).

Organic compounds (E.P. 244,830).—See XIII.

Film-forming composition. (U.S.P. 1,564,664).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of cellulose acetate silk. II. V. KARTASCHOV (Helv. Chim. Acta, 1926, **9**, 152—173; cf. B., 1926, 49).—By colorimetric measurements of the initial and final concentrations in the dye-bath the amount of various direct cotton dyes (purified by dialysis in tap water for 15 days and then in distilled water for 15 days) taken up by cellulose silk (celanese), and the velocity with which the dye is removed, have been determined, and, in the case of water-soluble dyes, compared with the partition coefficient of the dye between water and ethyl acetate. In many cases, *e.g.*, the phthaleins, the difference between the initial and final concentrations of the bath does not give a correct value for the amount of colour taken up by the fabric, since there is a precipitation of colour on the fibre surface, which makes difficult any sharp distinction between colloid suspensions and macrocrystalline suspensions. The results are opposed to the theory of Clavel, but support the theory that the dyeing of the silk is a phenomenon of solubilisation of the colour by the fibre by means of such groups as OH and NH_2 (Witt and others). Contrary to Clavel's theory it is found (*e.g.*, in the case of Eosine

I) that the free acid and its ethyl ester have greater dyeing powers than the sodium salt; and with water-soluble colours it is a general rule that the more soluble the dye and the smaller its partition coefficient between water and ethyl acetate, the less is its affinity for the fibre. The size of the particles is not important, as is shown by dry dyeing, and affects only the rate of diffusion into the interior of the fibre. In the case of insoluble colours it is shown, again contrary to Clavel's view, that a molecular dispersion will not dye the silk, but the finer the suspension the more rapid is the solubilisation of the colour. As an approximate rule it is found that if, in comparison with its molecular weight, the dye is easily soluble in ethyl acetate, it will dye artificial silk effectively. Colours developed on the fibre are mostly insoluble in water, and the rule for such colours applies here. The fibre in this case, however, becomes supersaturated with the dye, which crystallises out in the interior of the fibre and, owing to its insolubility, is not removed by washing. J. W. BAKER.

Colloid chemistry and printing. O. TREICHEL (Kolloid-Z., 1926, **38**, 80—81).—The principles underlying various printing processes are described. N. H. HARTSHORNE.

Growth of mould fungi on sizing and finishing materials. Growth of mould fungi on steeped wheat flour. MORRIS.—See V.

PATENTS.

Bleached and dyed furs. H. STEIN, W. E. AUSTIN, and I. LIEBOWITZ, Assrs. to STEIN FUR DYEING Co. (U.S.P. 1,564,378, 8.12.25. Appl., 3.5.24).—Cleaned fur is immersed in a solution of ferrous sulphate containing ammonium chloride, and is then bleached by treatment with hydrogen peroxide. The ferrous sulphate protects the skin against any harmful oxidation. T. S. WHEELER.

Treating [bleaching] vegetable fibres. P. THOMPSON (U.S.P. 1,565,602, 15.12.25. Appl., 10.3.24).—Vegetable fibre, *e.g.*, raw cotton, is shredded and soaked in a bleaching solution, such as a solution of hypochlorous acid, and then rapidly dried by the action of a large volume of hot dry air which is passed rapidly over it. T. S. WHEELER.

Preparing straw braid for bleaching. E. W. GEISLER, Assr. to R. H. COMEY BROOKLYN Co. (U.S.P. 1,568,664, 5.1.26. Appl., 15.6.25).—Straw braid is subjected while moist to chlorine gas before treatment with a bleaching agent other than chlorine. A. GEAKE.

Weighting, mordanting, and waterproofing animal and vegetable textile materials. J. SONNERY (F.P. 594,524, 1.12.23).—Textile material is impregnated with barium aluminate solution, and the aluminate is subsequently decomposed *in situ* by treatment with carbon dioxide, or with solutions of alkali or ammonium carbonates, phosphates, or silicates. Alternatively, impregnation with a solution containing, *e.g.*, ammonium carbonate may precede treatment with barium aluminate. The process may be repeated until the material is weighted to the desired extent. L. A. COLES.

Preparations for textile [sizing]. BRITISH CELANESE, LTD., C. F. RYLEY, and G. A. AWCOCK (E.P. [A] 244,947, and [B] 247,979, 27.6.24).—(A) A sizing composition suitable for celanese, and for other artificial and natural textile yarns, consists of a water-insoluble soap of resin acids or naphthenic acids and one or more lubricating agents, namely, non-volatile, non-drying oils, fats, waxes, or liquid or solid fatty acids. Soaps of calcium, magnesium, zinc, or aluminium are preferred. The composition is preferably prepared by dissolving the constituents in an indifferent volatile solvent, such as benzene, toluene, xylene, or oil of turpentine, and this solution is applied to the yarn. The amount of size is generally 5—10% of the weight of the yarn. This size is not brittle, dusty, or sticky. (B) A sizing composition suitable for celanese and other textile yarns consists of a mixture of one or more lubricating agents, such as non-drying oils, fats, waxes, or liquid or solid fatty acids, with one or more resins, such as gum mastic or dammar resin, which will form a non-sticky film on the yarn. Drying oils and colophony are unsuitable. The ingredients are preferably dissolved in an indifferent solvent, such as benzene, toluene, xylene, or oil of turpentine, and the solution is applied to the yarn. The size is not brittle, dusty, or sticky. Soaps of fatty acids may be added. A. GEAKE.

Size softener [for textiles]. J. W. MARSH and M. I. AISCHE (E.P. 245,199, 2.10.24).—A size softener is prepared by mixing a soap of a fatty acid, a hydroxy-fatty acid, or a sulpho-fatty acid, free or nearly free from resin acid, with hydrated silicates or substances containing silicates or silica, such as china clay, allophane, pyrophyllite, talc, steatite, pinite, fuller's earth, okenite, xonaltite, apophyllite, or silicic acid. Soaps of sodium, potassium, aluminium, magnesium, calcium, manganese, zinc, and the like, may be used, and a deliquescent salt may be added. The amount of silicious matter should not be more than six times the weight of the soap if the size contains a filler. A. GEAKE.

Dyeing and analogous treatment of yarns in wound form. J., T., and J. BRANDWOOD (E.P. 246,199, 16.10.24).

Mixtures containing finely-divided copper compounds (G.P. 419,460).—See XVI.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Chemistry of the lead chamber [sulphuric acid] process. J. FISCHLER (Z. angew. Chem. 1926, **39**, 55—56).—The use of sprays of dilute sulphuric acid in the chamber instead of steam and water (cf. Raschig, J.S.C.I., 1911, 172), was adopted by the author three years ago with good results.

The average concentration of the acid produced is improved, and the consumption of nitric acid reduced.
W. T. K. BRAUNHOLTZ.

Development of sulphuric acid manufacture from gases obtained by roasting lead ores. W. HEYER (Metall u. Erz, 1925, **22**, 439—452; Chem. Zentr., 1926, I., 464).—After the introduction of the Huntington–Heberlein process for the treatment of lead ores at the Friedrichshütte works, it was decided, though with great hesitation, to build a lead chamber sulphuric acid plant in order to prevent nuisance from the roaster gases. These contained 5·0—7·5% SO_2 by volume, up to 3·5% CO_2 and 50—136 g. of moisture per cb. m. They were delivered at a temperature of 290—320°. It was found necessary to heat them to 525° by passing through heated iron pipes. Difficulties arising during the first year's working are described. C. IRWIN.

Constitution of bleaching powder. B. NEUMANN and F. HAUCK (Z. Elektrochem., 1926, **32**, 18—31).—All previous researches on the constitution of bleaching powder are held to have failed, because pure starting materials were not used. When bleaching powder is kept for a long time in closed vessels, the percentage of available chlorine falls at first and then becomes constant at about 39·0% for completely chlorinated samples. Free calcium chloride is never present in pure fresh bleaching powder. The ratio of chlorinated to unchlorinated lime is 3:1. With samples exposed to the air the available chlorine falls continually, and finally only chloride chlorine is left. Samples in closed vessels exposed to sunlight undergo an intramolecular change as follows: $6CaOCl_2=5CaCl_2+Ca(ClO_3)_2$. The mechanically retained chlorine, which is always present in a freshly prepared specimen, may be completely removed within a half to one hour by a stream of nitrogen, dry air, or dry carbon dioxide, leaving a product containing always 39·0% of available chlorine. Here also the ratio of chlorinated to unchlorinated lime is 3 : 1. The optimum condition of chlorination is reached with calcium hydroxide containing 3·75—4·0% of excess water. The unsatisfactory agreement between the determinations of the heat of reaction in the chlorination of lime made by Neumann and Müller (B., 1925, 445) was due to the presence of varying amounts of mechanically retained chlorine. When the results are re-calculated for bleaching powder which is completely "blown," satisfactory agreement is obtained, viz., 101·55±0·7 cal. per g. of bleaching powder or 252·0±2·0 cal. per g. of chlorine gas. Moist carbon dioxide reacts with bleaching powder with formation of calcium chloride. The presence of calcium carbonate in the lime suffices to change the whole of the bleaching chlorine into calcium chloride. Chlorination under pressure does not result in the chlorination of all the lime. The somewhat higher result obtained is due to the formation, through the heat developed, of some chlorate and chloride. The formation of chlorate in bleaching powder commences at about 60° and proceeds most rapidly between 70° and 85°; above this decomposition occurs. If chlorination is carried out with exclusion of air at 65—70°, increasing quantities of chloride and chlorate are formed, always in the proportion 5 : 1. This is evidence that they are formed by an intramolecular change in the calcium chlorohypochlorite according to the equation given above. To show that bleaching powder actually contains the OCl and Cl groups arranged according to the Odling formula, the pure product has been heated in sealed tubes in the absence of air at 70—80°, resulting in the almost complete change to chloride and chlorate in the proportion 5:1. It is concluded that pure bleaching powder has the formula $3Ca(OCl)Cl, CaO,6H_2O$. From a practical point of view the chief conclusion to be drawn from the investigation is that the purity of the starting materials is of the greatest importance in determining the quality of the finished product.
N. H. HARTSHORNE.

Purification of "antichlor" [sodium thiosulphate] from sulphite and sulphate by crystallisation. L. WÖHLER and J. DIERKSEN (Z. angew. Chem., 1926, 39, 33—36).—Solubility isotherms are drawn for mixtures of sodium thiosulphate and sulphate, thiosulphate and sulphite, and the three together, in water. The solubility of sodium thiosulphate increases rapidly with rise of temperature, and is depressed but little by the presence of sulphate though more by sulphite. The curves illustrate how it is possible to obtain sodium thiosulphate free from sulphite and sulphate by crystallisation.
W. T. K. BRAUNHOLTZ.

Rate of solution and availability of commercial limes. R. T. HASLAM, F. W. ADAMS, and R. H. KEAN (Ind. Eng. Chem., 1926, **18**, 19—23).—The availability of a commercial lime, which determines its value as an alkali, depends on its rate of dissolution. It is deduced from Fick's law of diffusion and confirmed by experimental results with commercial high-calcium and high-magnesium limes, that the rate of solution increases with the fineness of the lime, increasing directly with the surface area exposed and with the ultimate solubility of the lime in the surrounding solution. Thus the solubility is increased in a solution containing an acid, such as hydrochloric acid, forming a soluble calcium salt, and diminished in a solution containing oxalic acid, owing to the formation of a crust of insoluble salt on the surface of the lime. Similarly the solubility is higher in a 2% sugar solution than in water, and lower in a solution of sodium carbonate. Any availability test for lime must therefore be based on the process in which the lime is to be used.
B. W. CLARKE.

Basic magnesium carbonates. G. R. LEVI (Giorn. Chim. Ind. Appl., 1925, 7, 697—698).—*X*-Ray analysis by the powder method shows that the crystalline and compact saccharoid varieties of normal magnesium carbonate, and also the dense, earthy form known as giobertite, have one and the same crystal structure. Basic magnesium carbonates, either prepared in various ways in the laboratory or

of industrial origin, have, in all cases, the composition and crystal structure of hydromagnesite, $4MgO,3CO_2,4H_2O$; the only other basic magnesium carbonate is artinite, $2MgO,CO_2,4H_2O$, which has not been reproduced artificially. The compound of the supposed formula, $11MgCO_3,3Mg(OH)_2,11H_2O$ obtained by Greider (B., 1923, 142 A) is identical with hydromagnesite, and a commercial trihydrated normal magnesium carbonate of German manufacture corresponds in structure with the mineral nesqueonite. T. H. POPE.

Haglund process for the electrothermic production of pure aluminium oxide. T. R. HAGLUND (Ind. Eng. Chem., 1926, **18**, 67—68).—A proportion of pyrrhotite or other heavy metal sulphide is added to the mixture of bauxite and carbon (usually coke) to be treated in an electric furnace, so that aluminium sulphide is formed. This produces a sulphide-oxide slag which is very fluid and easily separated from the reduced impurities of the bauxite (iron etc.). The alumina crystallises out from this slag on cooling, the size of the crystals being regulated by the rate of cooling; this is an important feature in the manufacture of abrasives. The slag is crushed and treated with water, the hydrogen sulphide evolved being used to convert part of the original bauxite into sulphide, whilst the alumina crystals are separated and washed. The product is purer than that generally used for the electrolytic production of aluminium, and the cost of production is less than of that obtained by the usual methods in Europe. B. W. CLARKE.

New method of preparing lead arsenates. L. CAMBI and G. BOZZA (Giorn. Chim. Ind. Appl., 1925, **7**, 687—696).—With the help of the electrometric method the authors have traced the course of the precipitation of the various lead and calcium arsenates by treating di- or tri-sodium arsenate solutions with lead or calcium nitrate or chloride solutions, both separately and together, and measured the velocity of the double exchange between tricalcium arsenate and soluble lead salts. The compositions are given of the lead arsenates precipitated in presence of calcium salts and of those obtained by the action of calcium hydroxide on mixtures of solutions of arsenic acid and of lead chloride or nitrate. Precipitates formed under the last conditions contain a practically negligible proportion of calcium oxide, and those produced in presence of a chloride consist principally of lead arsenate, together with a certain amount of chloroarsenates. Hence, in the manufacture of lead arsenate, preliminary removal of the hydrochloric acid from the arsenical liquors is not necessary. As regards their physical properties, the precipitates obtained by the above methods are quite suitable for use as insecticides. T. H. POPE.

Influence of ethylene on explosion limits of oxyhydrogen gas. JORISSEN and ONGKIEHONG.—See II.

Safety in manufacture of nitric, sulphuric, and mixed acids, and ammonium nitrate. KERSHAW.—See XXII.

PATENTS.

[Manufacture of] sulphuric acid. R. MORITZ (F.P. 593,627, 19.1.25. Conv., 30.1.24).—Dilute nitric acid is sprayed into the lead chambers in place of atomised water. C. IRWIN.

[Manufacture of] sulphuric acid. P. VEROLA (F.P. 594,078, 14.5.24).—Sulphur dioxide in the form of burner gas is passed over a catalyst, such as pyrites cinders. The unconverted sulphur dioxide in the gas so treated is absorbed by carbon or other suitable medium and returned to the process. C. IRWIN.

Manufacture of carbonyl compounds [iron carbonyl]. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 244,895, 26.11.24).—Iron carbonyl is prepared by passing carbon monoxide at a pressure above 50 atm. over reduced iron heated to 100—200° with a gas velocity sufficient to prevent any deposition of iron carbonyl on the metal. The exit gases contain 2—8% of iron carbonyl, $Fe(CO)_5$, by volume and this is condensed by cooling without reduction of pressure. The reaction chamber is lined with copper and has the form of a vertical cylinder with a suitable heating device. It is charged with burnt pyrites in small lumps. This is reduced by a current of hydrogen at ordinary pressure and a temperature of 500° and is then ready for treatment with carbon monoxide. The latter may be supplied in the form of water-gas, in which case a part of the reaction gases must be continuously replaced. To avoid decomposition of iron carbonyl it is essential to cool the gas below 140° before the pressure is reduced. C. IRWIN.

Method of making anhydrous magnesium chloride. P. COTTRINGER and W. R. CULLINGS, Assrs. to DOW CHEMICAL CO. (U.S.P. 1,567,317, 29.12.25. Appl., 1.4.21).—A rotary drum which dips beneath the surface of magnesium chloride solution is internally heated by means of gas. The products of combustion are passed over the surface of the drum in such a way as to bring the film of magnesium chloride to successively higher temperatures before it is removed. C. IRWIN.

Conversion of nitrides which are difficult to decompose, as silicon nitride, into an easily decomposed form. E. FRIEDERICH (G.P. 419,211, 15.8.24).—Silicon nitride is heated with oxides of the alkaline-earth metals or similar compounds. A mixture of silicon nitride and aluminium nitride, such as is obtained by heating natural silicates with carbon in a current of nitrogen, may also be used. The powdered nitride is heated with an excess of lime in a vacuum or an indifferent atmosphere to above 1000°. The presence of calcium chloride or calcium fluoride lowers the temperature necessary for the conversion. C. IRWIN.

Utilisation of residues from sodium sulphide manufacture. RHENANIA VEREIN CHEM. FABR. A.-G., Assees. of H. MENDHEIM (G.P. 419,285, 16.8.24).—The residues are lixiviated with a limited amount of water, and sulphur and sulphur dioxide (or sodium hydrogen sulphite) are added to the liquor in such proportions as to convert all alkaline sodium compounds present into thiosulphate, which is capable of crystallisation without previous evaporation. C. IRWIN.

Manufacture of chromium sulphate solution free from iron from ferrochrome. H. C. STARCK, KOMMANDITGES. AUF AKTIEN, F. KLAUS, and R. BASLER (G.P. 419,365, 12.8.23).—A solution of ferrochrome in sulphuric acid is treated with potassium dichromate and heated to the boiling point either at atmospheric pressure, preferably with addition of sodium carbonate, or at higher pressure. After separation of precipitated iron compounds, the solution of chromium sulphate may be mixed with potassium sulphate for the production of chrome alum, if necessary first reducing any ferric compounds still present with sulphur dioxide.
P. B. ROBINSON.

Manufacture of strontium carbonate. BADISCHE ANILIN- U. SODA-FABR., Assees. of J. FAHRENHORST, R. GRIESSBACH, and F. SANDER (G.P. 419,717, 16.10.23. Addn. to 336,767 ; B., 1921, 583 A).—The process for double decomposition of calcium sulphate and ammonium carbonate is applied to strontium sulphate. C. IRWIN.

Manufacture of thenardite and Glauber's salt. GEWERKSCHAFT BURBACH, and F. WIENERT (G.P. 421,325, 6.12.23).—Mixtures of kieserite (including the slightly soluble form) and rock-salt in suitable proportions, or alternatively potash-salt residues containing a suitable proportion of kieserite, are converted by double decomposition into van't-hoffite. This is separated by washing from undecomposed initial material and dissolved. The solution is saturated with rock salt and, when warm, thenardite separates out, whilst in the cold Glauber's salt is obtained. J. S. G. THOMAS.

Method of producing alumina. R. JACOBSSON (U.S.P. 1,570,353, 19.1.26. Appl., 25.8.24).—See E.P. 221,209 ; B., 1925, 758.

Manufacture of alkali-metal amides. T. EWAN (U.S.P. 1,570,467, 19.1.26. Appl., 31.3.24).—See E.P. 222,718 ; B., 1924, 980.

Synthesis of ammonia by means of catalysts. H. HARTER, Assr. to A. T. OTTO AND SONS (U.S.P. 1,570,485, 19.1.26. Appl., 11.2.25).—See E.P. 241,771 ; B., 1926, 12.

Colloids (G.P. 416,062).—See I.

Intimately mixing liquids, *e.g.*, **for causticising** (G.P. 419,812).—See I.

Electrolytic cells for manufacture of chlorates (G.P. 418,945).—See XI.

VIII.—GLASS; CERAMICS.

Use of white enamels containing antimony compounds [in the preparation of enamelled iron cooking utensils]. E. SVAGR (Chem. Listy, 1926, **20**, 21—24).—Tests of the amount of antimony dissolved from iron utensils enamelled with mixtures containing sodium metantimonate have shown that no antimony dissolves in any organic acid of the strength met with in foodstuffs. Prolonged use for cooking purposes of enamelled iron ware containing antimony opacifiers has resulted in no ill-effects. Antimonic compounds are much less toxic than antimonious. A. R. POWELL.

Relationship between soluble iron and colloids in certain clays. MCCARTHY.—See XVI.

PATENTS.

Production of articles from vitreous slags and glass refuse. F. NETTEL (E.P. 227,848, 15.1.25. Conv., 15.1.24).—A porous structure is given to articles cast from vitreous slag or glass refuse, by injecting into the molten slag or glass as it flows into the mould, vapour or gas mixed with some substance which generates gases or vapours on heating, *e.g.*, limestone or other carbonate. P. B. ROBINSON.

Control of the softening temperature of vitreous material. W. C. SPROESSER, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,565,598, 15.12.25. Appl., 17.10.22).—By treating the surface of glass with a solution of sodium ammonium phosphate the softening point of the glass is raised about 100°.
T. S. WHEELER.

Unburned refractory brick. R. YOUNGMAN, Assr. to HARBISON-WALKER REFRACTORIES CO. (U.S.P. 1,564,394, 8.12.25. Appl., 29.2.24).—60 pts. of calcined magnesite, 35 pts. of chrome ore, and 5 pts. of sodium silicate are mixed with water, moulded, and dried at about 100°. The product can be used directly in furnace construction without burning. T. S. WHEELER.

Glass. A. H. COMPTON, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,570,876, 26.1.26. Appl., 16.10.20).—See E.P. 170,563 ; B., 1922, 634 A.

Tunnel kilns. L. WILPUTTE (E.P. 245,827, 13.10.24).—See U.S.P. 1,522,166 ; B., 1925, 172. (Reference is directed in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 498 of 1881.)

Kiln and like structure, and heat treatment. N. LENGERSDORFF (U.S.P. 1,571,058, 26.1.26. Appl., 9.2.24).—See E.P. 236,229 ; B., 1925, 882.

Drawing and flattening sheet glass. W. J. TENNANT. From EMPIRE MACHINE CO. (E.P. 246,057, 21.8.25).

Resinous substances for optical apparatus (G.P. 416,753).—See XIII.

IX.—BUILDING MATERIALS.

Setting of hydraulic cements. Baykoff (Compt. rend., 1926, **182**, 128—129).—The explanation advanced by Le Chatelier for the setting of cements is considered to be insufficient. It is suggested that the setting takes place in three stages: (1) dissolution, during which the liquid is progressively saturated with the different soluble substances; (2) "colloidation," during which the products of the chemical reaction are obtained in the colloidal state, this stage corresponding with the initial set; (3) crystallisation, in which the gels are transformed into aggregates of large crystals, on account of the greater solubility of smaller crystals. This theory was verified by shaking Portland cement with water and analysing the liquid from time to time, and also analysing the crystals (calcium hydroxide) which formed gradually on the walls of the containing vessel following an initial separation of flocculent material.
L. L. Bircumshaw.

Relation between water permeability and water absorption of concrete. E. Anderson (Ind. Eng. Chem., 1926, **18**, 17—18).—There is no direct relation between the water absorption, determined by the gain in weight when a dry concrete specimen is saturated with water, and the water permeability. The water absorption as generally determined is not a true measure of the porosity, since a certain amount of water is taken up by colloidal substances partially dehydrated by drying at 110°, in addition to the water to fill the capillary pores of the concrete. The water permeability is best measured by the weight of water passing through unit area in unit time under a definite pressure, thus determining the actual resistance to flow of water, or water-tightness. B. W. Clarke.

Patents.

Production of cement in blast furnaces. Gewerkschaft Lutz III. (E.P. 228,128, 2.1.25. Conv., 21.1.24).—Slag with a high percentage of lime is formed in a blast furnace and kept in a fluid state by a preheated air blast enriched with oxygen, thus producing simultaneously iron and a slag, which on grinding is used as a cement. Similarly with iron ores containing argillaceous earth, a slag, with a high percentage of alumina is formed, producing a rapid-hardening aluminous cement on grinding.
B. W. Clarke.

Simultaneous production of cement and combustible gas. Gewerkschaft Lutz III. (E.P. 228,129, 2.1.25. Conv., 22.1.24).—Bituminous fuel is burnt in a producer with a mixture of chalk and clay, by means of an oxygen-enriched air-blast, so that a gas of high calorific value is produced together with a fluid slag which is drawn off and ground for use as cement. An aluminous cement is produced if iron ore is added so that the silica of the slag is fixed as ferrosilicon. B. W. Clarke.

[Cellular] concrete. K. P. Billner (E.P. 244,693, 11.3.25).—Aluminium or zinc powder in the proportion of 0·03—0·2% is intimately mixed with cement in a grinding or mixing mill, so that the metallic powder adheres to the particles of cement. The product may be kept for a considerable period without losing the property of forming "expanded" concrete when mixed with water and lime, and is suitable for transporting etc., for the production of cellular concrete cast *in situ*. B. W. Clarke.

Waterproof Portland cement. C. N. Miller (U.S.P. 1,566,498, 22.12.25. Appl., 26.2.24).—A mixture of β-naphthol and non-volatile oil is incorporated with the cement. B. W. Clarke.

Unfused aluminous cement. R. Décolland (Addn. 29,278, 29.1.24, to F.P. 573,517; cf. E.P. 211,497; B., 1925, 208).—The process described in the chief patent is modified by adding a small amount of sodium chloride or a similar salt as a flux to the red-hot mass in the kiln, and by lengthening the fixed head of the rotary kiln to form a separate chamber. B. W. Clarke.

Magnesia-zinc cement. L. D. Legentil and R. Greffe (F.P. 591,414, 6.1.25).—A mixture of magnesia in magnesium chloride solution and zinc oxide in zinc chloride solution, with or without the addition of borax or hydrochloric acid, is thoroughly incorporated with a filler, such as sawdust, sand, limestone dust, etc., forming a cement which sets very quickly and is harder and more water-resistant than Sorel (magnesia) cement. B. W. Clarke.

Mortar for cement. A. Sommer (G.P. 406,911, 22.5.20).—Portland cement or a similar hydraulic cement, or a mixture of limestone and clay, or raw cement mixture, is treated with hydrochloric acid, or heated with calcium or magnesium chloride, and mixed with bitumen, oil, or the like to form a mortar for cement etc. B. W. Clarke.

Grinding blast-furnace cement or slag cement. R. Grün (G.P. 418,355, 2.12.23).—Portland cement clinker or sintered calcareous marl, added to the slag cement as an improver, is subjected to a preliminary fine grinding. This results in a more even distribution of the clinker and gives an improved product. B. W. Clarke.

Wood preservation. C. T. Henderson and L. Rosenstein (U.S.P. 1,565,503, 15.12.25. Appl., 21.6.22).—10 pts. of a 70% solution of zinc chloride are mixed with 85 pts. of a heavy hydrocarbon oil, such as asphalt, and the mixture is emulsified by addition of 5 pts. of an emulsifying agent, such as naphthalenesulphonic acid. The product preserves wood and renders it waterproof.
T. S. Wheeler.

Treatment [mineralising] of fibrous materials. J. R. Garrow, Assr. to Novocrete and Cement Products Co., Ltd. (U.S.P. 1,571,048, 26.1.26. Appl., 28.8.24).—See E.P. 220,677 and 225,912; B., 1925, 12, 134.

Manufacture of artificial wood [from sawdust and cement]. C. D. Burney (E.P. 245,812, 14.8.24).

Preservative (G.P. 419,097).—See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Passivity of iron by dilute nitric acid. T. FUJIHARA (Ind. Eng. Chem., 1926, **18**, 62—63).—A protective film on iron has been produced by immersion in a 6% solution of nitric acid in alcohol. This oxide film, which is similar to that produced by the action of chromic acid, is very resistant to corrosion by water, particularly in the absence of carbon dioxide. The action of the acid produces ferrous oxide, which reacts with water yielding ferrous hydroxide. The dissolved ferrous hydroxide being alkaline renders the iron passive. To obtain a satisfactory protection alcohol is essential in making the etching solution. The concentration of acid may vary between 2 and 10%. M. COOK.

A2 line in the equilibrium diagram of the iron–carbon system. K. HONDA (J. Iron and Steel Inst., 1925, **112**, 345—348).—In the equilibrium diagram of the iron–carbon system (B., 1922, 418 A), the A2 line is shown as a dotted line falling at first from 790° to 770° and then remaining nearly horizontal. Magnetic analysis shows the distinction between the A1 and A2 transformations, and beyond the A1 point weak magnetisation is observable for hypoeutectoid and even for hyper-eutectoid steels. Considered theoretically the A2 line may be divided into three parts. One portion represents the critical temperature at which austenite changes from a weakly magnetic to a paramagnetic substance ; a second portion represents a change in concentration of austenite. In steels consisting of a mixture of ferrite and austenite the third portion must consist of two horizontal lines about 20° apart, the upper one correctly called the A2 line, the lower one, including also the previous two portions, being termed the A′2 line for differentiation. These may only be introduced into the diagram as dotted lines, having no connexion with the lines representing change of phase. C. A. KING.

Strain detection in mild steel by wash coating. R. S. JOHNSTON (J. Iron and Steel Inst., 1925, **112**, 341—343).—The visual detection of strain lines in steel is very sensitive if the steel is coated with a thin wash of Portland cement. Full-sized structural forms may be examined if treated in this way, and it was noted that small irregularities in a plate, such as a small hole or even a stamped identification mark, became the centre of strain effects and showed radiating strain lines before the remainder of the plate developed the corresponding stress.
C. A. KING.

Brittleness of cold-worked steel. G. DELBART (Compt. rend., 1926, **182**, 131—133).—The brittleness of four different samples of cold-worked steel was studied by means of impact tests (Charpy apparatus). The influence of annealing at various temperatures between successive periods of cold-work was also studied, and microscopical examination showed that in steels annealed below 700°, the cementite had coalesced, whilst those annealed between 700° and 850° showed a mixture of sorbite and globular pearlite, and those annealed above 850° showed only sorbitic pearlite. L. L. BIRCUMSHAW.

Iron–iron sulphide alloys. B. BOGITCH (Compt. rend., 1926, **182**, 217—219).—Electrolytic iron–iron sulphide mixtures fused in a crucible of refractory earthenware and protected from oxidation by sodium silicate do not form two liquid phases. The presence of oxygen or copper produces a heterogeneous mixture, and if the latter exceeds 3%, two layers are formed. Carbon causes the separation of two liquid phases, the upper of constant sulphur-content, and the lower containing an amount dependent on the carbon-content. An explanation of the desulphurising of steel is thus suggested.
J. GRANT.

Properties of heat-treated nickel-chrome steels. L. GUILLET (Compt. rend., 1926, **182**, 249—251).—Specimens of nickel-chrome steel were quenched in oil from 900°. One series was then reheated to 650°, cooled very slowly (35 hrs.) from 650° to 410°, and then cooled in air to room temperature, after which it was again heated to 650° and cooled in air (treatment A). For a second series the two reheatings were in the reverse order (treatment B). At room temperature the two series had almost the same tensile strength, but those submitted to treatment B were much more brittle, less resilient, and less resistant to repeated impact. On testing at 200°, series B were no longer brittle, but it was necessary to heat to 500° before cooling in air gave a specimen comparable to series A. The brittleness is thus the result of very slow cooling, and some kind of transformation occurs at about 200°, but no volume, micrographic, or thermo-electric change could be detected. The steels contained C 0·26%, Ni 3·76%, Cr 0·67%, Mn 0·54%, Si 0·11%, S 0·037%, P 0·021%. W. HUME-ROTHERY.

Zinc distillation in vertical retorts fired with lignite producer gas. C. ROTT (Feuerungstech., 1926, **14**, 85—86).—A description, with reference to plans, is given of a furnace for the distillation of zinc from ores in vertical retorts, 16 of which replace the usual 32 horizontal retorts in a furnace of the normal size. The retorts consist of oval fireclay tubes with movable bottoms through which the residues may be discharged into wagons running below the furnace so that no zinc fumes escape into the air. The upper ends of the retort are funnel-shaped to take a bevelled lid which may be readily removed for charging the retorts from tilting wagons running along the top of the furnace. Along the middle of the furnace below the retort level a large mixing chamber is provided in which preheated air and producer gas from lignite are mixed and from which the gas passes into the combustion chambers around each retort. The producer gas inlet flue is below and the air-preheating flues are on either side of the mixing chamber; the flues for removing the burnt gases are on either side of the gas inlet flue. The condensers, which are larger than those usually employed owing to the

retort charge being more than twice as large, project just inside the upper part of the retorts. The furnace is more easily operated, requires less labour in recharging and discharging the retorts and in the manufacture and replacement of retorts, and has lower running costs and a larger output than that with horizontal retorts. A. R. POWELL.

Rapid detection of small amounts of aluminium in certain non-ferrous materials [alloys]. G. E. F. LUNDELL and H. B. KNOWLES (Ind. Eng. Chem., 1926, **18**, 60—61).—Aurintricarboxylic acid can be used to detect aluminium in an approximately quantitative way when the percentage present is less than 0·05. To 1 g. of alloy dissolved in 5 c.c. of concentrated nitric acid are added 30 c.c. of an 8% solution of caustic soda. After boiling for 1 min., 20 c.c. of an 8% solution of sodium sulphide are added, the solution is filtered, the filtrate acidified with 1 : 1 hydrochloric acid, and 2 c.c. of the acid are added in excess. The mixture is digested at 40—60° until the precipitate settles, filtered, hydrogen sulphide expelled from the filtrate, and the solution clarified (if sulphur is present) with nitric acid, evaporated to 20—30 c.c., and filtered if necessary. 10 c.c. of 36% acetic acid and 5 c.c. of 0·2% solution of aurintricarboxylic acid are added, and then a 10% solution of ammonium carbonate in dilute (1 : 2) ammonia, with stirring, until an excess of 5—10 c.c. is present. The colour of the solution or the amount of separated lake is then compared with standards. If less sodium sulphide is added and the second filtration omitted, the time is shortened to 5—10 min., and the colour should not be obscured if not more than 10% of tin or 25% of lead were present in the alloy, whilst with such alloys as brass and those giving large precipitates with sodium hydroxide no sulphide need be used. D. G. HEWER.

Restraint of exaggerated grain growth in critically strained metal. G. L. KELLEY and J. WINLOCK (J. Franklin Inst., 1926, **201**, 71—77).—Critically strained sheet steel and aluminium exhibit exaggerated grain growth under normal conditions of annealing. Experiments were carried out to determine whether a preliminary heat treatment below the recrystallisation temperature would inhibit exaggerated growth when the metal was subsequently annealed at higher temperatures. Results were inconsistent but indicated that such treatment was sometimes effective. C. J. SMITHELLS.

Determination of small quantities of selenium in sulphide minerals. M. SCHMIDT (Metall u. Erz, 1925, **22**, 511—512; Chem. Zentr., 1926, I., 739).—The mineral is dissolved in *aqua regia* and the solution evaporated with sulphuric acid until copious fumes are evolved. After cooling, water is added and the insoluble material filtered off. The filtrate is saturated with hydrogen sulphide and the washed precipitate digested with ammonium carbonate for the removal of arsenic. The remainder of the sulphide precipitate is dissolved in *aqua regia*, and the gold, platinum, and selenium are precipitated from the solution by means of stannous chloride solution (100 g. of stannous chloride dissolved in 1 litre of concentrated hydrochloric acid). For quantitative work, in the absence of gold and platinum, the colour of the colloidal selenium solution so produced is compared with that of a standard solution of selenious acid similarly treated. The limit of sensitiveness of the method is 0·025 mg. of selenium. A. R. POWELL.

Volatile tin compounds and tin poisoning. W. VAUBEL (Münch. med. Wochenschr., 1925, **72**, 1161; Chem. Zentr., 1926, I., 336).—Dissolution of tin in hydrochloric acid produces a gas that has a peculiar metallic odour sometimes resembling the odour of stewed meat. The gas burns with a luminous flame, gives a mirror of metallic tin, and reacts with silver nitrate solution, indicating that it contains a volatile tin compound such as stannous chloride or hydride or a mixture of these. The stannous compounds are only slightly poisonous, but are readily oxidised by the air to the much more poisonous stannic compounds. (Cf. Vaubel, B., 1924, 560; Paneth and Joachimoglu, Ber., 1925, **57**, 1925.) A. R. POWELL.

Sulphuric acid from gases obtained by roasting lead ores. HEYER.—See VII.

Electrothermic production of aluminium oxide. HAGLUND.—See VII.

Mosaic gold. HADERT.—See XIII.

PATENTS.

Iron castings. J. E. FLETCHER and H. J. YOUNG (E.P. 245,196, 1.10.24 and 18.3.25).—Iron castings produced in the first instance with a predominating pearlitic structure are heated to above the Ac1 point and cooled quickly to give an austenitic structure. The castings are then reheated to a temperature below the Ac1 point and are cooled more slowly to produce a mixed structure of martensite, sorbite, and troostite. The mixed structure may be obtained after the first heating if the rapid cooling is arrested at a suitable temperature. Metal for casting may contain small quantities of nickel, cobalt, molybdenum, tungsten, vanadium, boron, or chromium. C. A. KING.

Direct production of iron and steel. R. H. M. L. TOURNIÉ (E.P. 245,223, 13.10.24).—A mixture of iron ore, flux, and carbonaceous matter is melted under oxidising conditions in a tilting furnace, the slag being removed periodically, until a bath of molten iron oxide containing reduced iron is produced. The iron oxide is then reduced and carburised by the action of reducing gases, either in the tilting furnace or in a separate converter. C. A. KING.

Annealing steel sheets. J. T. HAY, Assr. to UNITED ALLOY STEEL CORP. (U.S.P. 1,567,143, 29.12.25. Appl., 19.1.25).—Chrome steel sheets are annealed by heating in an oxidising atmosphere to form on them a soluble scale. M. COOK.

[Making] rustless iron and similar alloys. F. M. BECKET (U.S.P. 1,567,898, 29.12.25. Appl., 21.2.25).—In the production of corrosion-resisting

chromium alloys a low-carbon alloy containing more than 5% Cr and more than 40% Si is made in an electric furnace and treated with a metal oxide to oxidise silicon. M. COOK.

Recovering iron and sulphur from sulphide ores. H. CONDER (U.S.P. 1,567,916, 29.12.25. Appl., 20.3.23).—Crushed ore is heated in a closed retort into which hydrogen is passed to convert the sulphide ore into ferrous sulphide, sulphur being recovered from the hydrogen sulphide produced. M. COOK.

Dephosphorising iron and steel. I. M. SCOTT and S. PEACOCK (U.S.P. 1,568,186, 5.1.26. Appl., 7.3.25).—Oxidised compounds of chromium are added to a molten bath of basic iron or steel, in quantity sufficient to dephosphorise the greater part of the iron phosphide present in the metal. C. A. KING.

Rail steel. H. J. FORCE (U.S.P. 1,568,822, 5.1.26. Appl., 17.7.25).—Steel suitable for rails contains C 0·30—0·85%, Mn 1·15—1·90%, P not more than 0·05%, Si not more than 0·3%. C. A. KING.

Producing ferrozirconium. R. H. MCKEE, Assr. to J. G. DONALDSON (U.S.P. 1,565,280, 15.12.25. Appl., 28.7.21).—Zirconium oxide and ferrous sulphide are heated to 1500°, when sulphur dioxide is evolved and ferrozirconium is formed. T. S. WHEELER.

Manufacture of low-carbon iron–chromium alloys [stainless iron]. AKTIEBOLAGET FERROLEGERINGAR (G.P. 418,627, 18.8.23. Conv., 26.7.23. Addn. to 407,249).—Low-carbon iron alloys containing 8—30% Cr and less than 0·1% C are made by pouring molten iron low in carbon into molten ferrochromium low in carbon, which is melted in a separate electric furnace, using electrodes of low-carbon ferrochromium or of low-carbon iron. A. R. POWELL.

Utilisation of blast-furnace dust, burnt pyrites residues, and other ferruginous waste materials. A. SCHREGER (G.P. 418,101, 21.3.23 ; F.P. 578,774, 15.3.24).—The finely-divided raw material, mixed with a stream of air containing finely-divided fuel, is burnt in a chamber provided with a sloping floor, so that a liquid slag is formed, which is drawn off, broken up, and worked for iron in the usual way. B. W. CLARKE.

Utilisation of flue dust, burnt pyrites, or other ferruginous materials. A. SCHREGER (G.P. 419,068, 12.2.42. Addn. to 418,101 ; preceding).—The heat absorbed by the reduction of the ferruginous material is made up by introducing into the layer of glowing carbon at the lower part of the combustion chamber sufficient solid carbonaceous fuel together with the necessary cold or preheated air, and the height of the carbon layer is so regulated that during the passage of the molten charge through it complete reduction of the iron oxide takes place. A. R. POWELL.

Core for steel castings. ECKERT, OPPELT U. Co. (Austr. P. 100,746, 2.11.22. Conv., 5.12.21).—A core for steel castings consists wholly or partly of aluminium nitride or material containing aluminium nitride, together with the usual binders, such as molasses or tar. The use of this core prevents the burning of the interior of the casting, and the core does not sinter to the casting and is easily removed. A. R. POWELL.

Electrolytic process for removal of rust and scale from metals. SEAL CO. (LONDON), LTD., J. JASCOURT, H. D. PATTINSON, and J. ROSE (E.P. 245,170, 30.3.25).—Rust and scale may be removed from a metal by subjecting it to the preliminary process described in E.P. 131,455 (B., 1919, 779 A), and subsequently treating it as an anode in sea water or a prepared solution of similar composition or in a solution of a halogen salt or salts of one or more of the alkali metals. A. R. POWELL.

[Roasting] kilns. G. BALZ (E.P. 245,348, 6.6.25).—In a multideck roaster with alternate rotary and fixed hearths, the rotating hearths preferably rotate alternately in opposite directions, and the main stream of roasting gases passes upwards from a combustion device at the bottom over all the hearths in counter-current to the falling ore. In addition, smaller streams of cold air are admitted through the walls of one or more of the fixed hearths and impinge on the ore as it falls through the transfer ports from the hearth above. B. M. VENABLES.

Apparatus for hot galvanising. W. G. IMHOFF, Assr. to W. S. SMITH (U.S.P. 1,566,749, 22.12.25. Appl., 16.4.23). — Aluminium borosilicates are resistant to the action of molten zinc and may be used as an enamel to line vessels to be used in hot galvanising. T. S. WHEELER.

Alloying rare metals [tungsten and uranium]. W. B. GERO, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,566,793, 22.12.25. Appl., 14.6.23).—Finely divided tungsten and uranium are shaken together in a solution of paraffin oil in ether and are then sintered together. Uniform distribution of the metals is obtained and the finely-divided material is protected from oxidation. T. S. WHEELER.

Metal-melting method. W. C. WILLIAMS, Assr. to MCLAUGHLIN COAL REDUCTION CO. (U.S.P. 1,567,219, 29.12.25. Appl., 13.10.23).—Metal is melted in an open crucible supported on a stand within a cubical chamber by the heat of a pulverised-fuel flame directed circularly around the crucible and over the top. The flue outlet is directly above the crucible and is baffled, so that the incombustible material in the flame drops on to the surface of the metal in the crucible. The pulverised fuel used is ground so fine that at least 25% passes through a screen having 800 meshes to the linear inch and the air pressure used is less than 0·5 oz. per sq. in. A. R. POWELL.

Making metallic magnesium. P. COTTRINGER and S. B. HEATH, Assrs. to DOW CHEMICAL CO.

(U.S.P. 1,567,318, 29.12.25. Appl., 21.5.23).—Molten magnesium chloride is electrolysed in a closed bath, fresh quantities being introduced through a feed chamber surrounding the anode. C. Irwin.

Soldering alloy. E. D. Schwalm (U.S.P. 1,567,396, 29.12.25. Appl., 29.4.25).—A solder for aluminium is made by melting copper and tin with sodium and adding zinc to the melt. M. Cook.

Plated article and its manufacture. J. A. Smith (U.S.P. 1,567,625, 29.12.25. Appl., 23.1.25).—The article is first coated with a metal having a low melting point, then with a metal having a substantially higher melting point. The article is then heated to a temperature sufficient to cause alloying to take place between the base metal and the intermediate layer without the latter alloying with the outer layer. A. R. Powell.

Non-poisonous electro-plating baths. F. Halla (Austr.P. 101,016, 18.9.24).—Compounds of cyanamide or its polymerides are used, instead of cyanides, in electroplating baths and similar solutions, ions capable of forming complex compounds, such as thiosulphate and thiocyanate ions, being added, if necessary, to render the compounds soluble.
L. A. Coles.

Bearing metal. J. Karafiat (U.S.P. 1,568,224, 5.1.26. Appl., 1.2.24).—A bearing-metal alloy comprises one of the alloys in the lead–tin–antimony system, the composition of which is represented by a point in the ternary diagram within the triangle formed by joining the points corresponding with the following compositions :—(*a*) 10% Sn, 10% Sb, 80% Pb, (*b*) 55% Sn, 5% Sb, 40% Pb, (*c*) 50% Sn, 50% Sb, together with a small amount of any metal or metals, except thallium, that form a solid solution with tin and lead. A. R. Powell.

Bearing metal. C. L. Ackermann (F.P. 591,519, 10.1.25).—A bearing metal consisting chiefly of lead, with some antimony and less than 4% of arsenic, and cobalt or iron, or both, with smaller amounts of phosphorus, tin, or nickel, is harder and more resistant to compression than similar alloys containing tin in the place of arsenic.
B. W. Clarke.

Amalgamating means. C. C. Hamill (U.S.P. 1,568,276, 5.1.26. Appl., 12.7.24).—An amalgamating plate for the recovery of gold is coated with a mixture of mercury and silver filings to produce a rough surface. A. R. Powell.

Recovering copper [from ores]. J. W. Neill, Assr. to G. Matsukata (U.S.P. 1,568,464, 5.1.26. Appl., 20.4.25).—The ore is leached with an acid solution to dissolve the copper, which is subsequently precipitated by bringing the pregnant solution into contact with an aggregate consisting of coke having iron distributed throughout the mass. The resulting mixture of coke and copper is smelted.
A. R. Powell.

Purification of highly oxidisable metals [*e.g.*, uranium]. R. W. Moore, Assr. to Gen. Electric Co. (U.S.P. 1,568,685, 5.1.25. Appl., 2.3.23).—Uranium may be separated from its less fusible oxide by melting the metal in an inert atmosphere and allowing the fused metal to flow away from the oxidised portion. The process is preferably carried out in an induction furnace of the Northrup type operated in a vacuum. A. R. Powell.

Deoxidising metals not affected by carbon. E. Heuse (G.P. 413,691, 27.2.24).—Finely-divided carbon, in the form of soot or other pure form of carbon free from adsorbed gases, is mixed with, or spread over, the protective oxide layer on the surface of the molten metal. The process is applicable principally to metals with a low vaporisation point, where a high degree of purity is required and excess of the reducing agent must be avoided, *e.g.*, aluminium, magnesium, zinc. B. W. Clarke.

Recovery of tin from alloys containing iron. F. Wüst (G.P. 417,459, 11.11.24).—The alloy is melted with aluminium, silicon, or ferrosilicon which alloys with the iron and liberates the tin. Manganese, carbon, and the like may be added in order to reduce the m.p. of the iron alloy formed.
A. R. Powell.

Production of thick adherent deposits of electrolytic nickel on aluminium. Langbein Pfanhauser-Werke A.-G. (G.P. 418,923, 11.10.24).—Thick, adherent coatings of nickel may be deposited on articles made of aluminium or its alloys from the usual nickel baths if the temperature is kept below 25° and the current density between 3 and 5 amp. per sq. cm., while the article is maintained in rapid motion relative to the electrolyte until the desired thickness is obtained. A. R. Powell.

Manufacture of hard alloys [of cobalt, chromium, and tungsten]. Siemens u. Halske A.-G., Assees. of B. Fetkenheuer (G.P. 419,388, 2.11.21).—Extremely hard alloys are obtained by fusing in the electric furnace a mixture of tungsten or cobalt with carbon and subsequently adding chromium and cobalt or tungsten. The alloys may also be made by reducing one or all of the oxides of the metals with the required amount of carbon to leave sufficient in the alloy to harden it, and adding, when necessary, the remainder of the metals.
A. R. Powell.

Aluminium alloy wire. Aluminium-Ind. A.-G. (Swiss P. 110,527, 21.5.24).—Aluminium alloys containing components, such as silicon, copper, and magnesium silicide, which form solid solutions in aluminium, are rolled or pressed into stout wire which is heated nearly to the solidus temperature, quenched, drawn down further to the desired dimensions, and finally aged below 250°, preferably at 175—180°. Ageing causes the conductivity of the wires to return to that of pure aluminium whilst the tensile strength is about 80% greater, so that they are suitable for use as free conductors.
A. R. Powell.

Leaching process for extraction of metals. W. G. Perkins, Assr. to Metals Production Co. of North America (U.S.P. 1,570,858, 26.1.26. Appl., 25.7.25).—See E.P. 243,075; B., 1926, 96.

Apparatus for charging circular sintering pans. H. G. Torulf (E.P. 240,460, 22.9.25. Conv., 24.9.24).

Producing cement in blast furnaces (E.P. 228,128).—See IX.

Heating processes and apparatus utilising atomic hydrogen (E.P. 237,898).—See XI.

Silver from precipitates in photographic solutions (F.P. 591,546).—See XXI.

XI.—ELECTROTECHNICS.

New carbon electrode. C. Becker (Stahl u. Eisen, 1926, **46**, 44—46).—A carbon electrode which does not easily fracture, and will bear a load of 250 volts without burning away too rapidly, consists of a hardened (fired) core, along the outer surface of which run metallic conductors and an outer electrode of compressed unfired material, surrounded and supported by a metal gauze cylinder. This outer metal gauze cover may be double and enclose a layer of some refractory material, such as asbestos. The metallic conductors between the core and the outer electrode are connected to the leads bearing current to the electrode. The resulting distribution of current over the cross section, whereby the core carries the greater part, is necessary for the satisfactory working of large electrodes. The relatively light load borne by the outer electrode makes it possible to construct the latter easily and directly from the raw material. A. B. Manning.

New electrical insulator. A. Samuel (Compt. rend., 1926, **182**, 206—208).—By the action of sulphur chloride on a viscous condensation product of formaldehyde and cresol, with subsequent purification by solution and precipitation, an odourless white powder (thiolite) has been prepared; 12% of sulphur chloride is required, the hydrochloric acid being neutralised as produced. Heat with pressure produces softening followed by quick polymerisation, and a hard, infusible, uninflammable and permanent substance results, having high insulating properties, ($R=300\times10^6$ megohms/cm.; $\epsilon=4\cdot5$). A solution of thiolite in an organic solvent may be used as an insulating varnish, the coating being subsequently polymerised by heat. J. Grant.

Purifying used [transformer and switch] oils with fuller's earth. H. von der Heyden and K. Typke (Elektrotechn. Z., 1925, **46**, 1518—1520; Chem. Zentr., 1926, I, 545).—Attempts to neutralise organic acids in used oils with ammonium carbonate were unsuccessful. Stirring with fuller's earth is effective if the acid value is not high. A. Geake.

Electrothermic production of aluminium oxide. Haglund.—See VII.

Patents.

Heating processes and apparatus [utilising atomic hydrogen]. Brit. Thomson-Houston Co., Ltd., Assees. of I. Langmuir (E.P. 237,898, 22.7.25. Conv., 30.7.24).—Hydrogen is dissociated into atomic hydrogen by means of an electric arc, and the heat of recombination of the atoms (stated to be about 42,000 cals. per g.) is utilised to heat the work, thus avoiding the intense local heating of the plain arc, with the secondary advantage of a reducing and refining atmosphere. Descriptions are given of apparatus suitable for melting scrap, for welding, and for brazing, the heat in each case being provided by one or more units each consisting of an electric arc with inclined electrodes, with a jet of hydrogen within the angle of the electrodes and impinging on the work. The arc in hydrogen between $\frac{3}{8}$-in. electrodes, preferably of tungsten, operates at a high voltage, say 100 volts, and if the stream of hydrogen is increased so as to expand the arc even 600 volts may be used. In addition to the jet of hydrogen which becomes dissociated, additional "bathing" hydrogen is useful in some cases, *e.g.*, for the complete elimination of air or for spreading the heat over a large area as in brazing. B. M. Venables.

Withdrawal of vapours and gases from electrolytic tanks. W. A. F. Pfanhauser, and Langbein-Pfanhauser-Werke A-G. (E.P. 245,314, 20.3.25).—By means of a suction-pipe connected to an exhauster, vapours and gases are drawn into holes in the under surface of a hollow frame extending across the electrolytic tank above the liquid level. The frame supports the electrodes and carries the leads. J. Grant.

Electrolytic cell for the manufacture of chlorates. Verein für Chem. und Met. Produktion (G.P. 418,945, 8.10.23).—The cell, in which chlorates are produced direct, has the electrodes submerged below a relatively tall column of quiescent liquid. The current connexions to the anode or to the cathode, or to both, do not pass through this column of liquid. The graphite anodes are provided above and below with graphite contact pieces for connexion to the current leads. C. Irwin.

Composition for electric insulators. A. L. Labbe, Assr. to Amer. Smelting and Refining Co. (U.S.P. 1,567,277, 29.12.25. Appl., 16.8.21).—Insulating material for high-voltage electric current is formed by moulding and baking a mixture of not less than 40% of slaked lime free from magnesia, with Portland cement and sand. P. B. Robinson.

Body of high electron and light emission. C. A. Laise, Assr. to Electron Relay Co. (U.S.P. 1,569,095, 12.1.26. Appl., 21.11.23).—A body of high electron- and light-emission is composed of an intimate mixture containing not less than 90% of tungsten, 0·50—5·0% of thorium, not more than 0·50% of vanadium, and 0·55—2·50% of yttrium oxide. The finely powdered materials are compacted by

heat and pressure into a homogeneous coherent billet, which is hammered at a high temperature. An envelope consisting of a refractory oxide is formed on the billet, which is annealed and made ductile and finally drawn to the desired diameter.

J. S. G. THOMAS.

Electrodes for electric arc furnaces. INTERNAT. GENERAL ELECTRIC Co., Assees. of B. LÜDKE (E.P. 325,596, 13.6.25. Conv., 14.6.24).

See also pages 176, **Protecting metallic surfaces from deposition of scale** (U.S.P. 1,568,728). 176—7, **Electrical precipitation** (U.S.P. 1,569,492, 1,569,529, 1,569,595, and 1,569,597 and G.P. 421,151 and 421,259). 183, **Cracking oils** (U.S.P. 1,568,016). 196, **Removing rust and scale from metals** (E.P. 245,170). 204, **Concentrating or coagulating rubber emulsions** (E.P. 245,177). 206, **Removing acids from leather** (Austr. P. 99,929. 222, **Determining the bacterial content of air** (G.P. 420,725).

XII.—FATS; OILS; WAXES.

China wood [tung] oil. M. TOCH (J. S. C. I., 1925, **44**, 511—512, 517—518, 527—528T).—Tung oil is derived from *Aleurites cordata* grown principally in China, The roasting of the seeds and subsequent pressing are of a primitive character, with no care to keep the oil clean, hence the great variability in the characters of the oil, quite apart from adulteration. The terms Canton, Hankow, and Hong Kong oils are misnomers, these cities being simply collecting points. Sp. gr., dispersion, and refractive index are the most important constants when it is desired to detect adulteration.

The crude oil has a cloudy appearance, yellow colour, and characteristic odour. Two samples received direct from Hankow had the following constants : $d^{15\cdot5}$, 0·9399, 0·9408 ; $n_D^{21\cdot5}$, 1·5176, 1·5176 ; dispersion value (21·5°), 0·02025, 0·02032 ; heat test (Browne's), $11\frac{1}{4}$ min., 11 min.

For the past 15 years successful attempts have been made to grow the tung oil tree in U.S.A. and the resulting oil is more uniform and paler in colour than that from China. The American oil polymerises more rapidly, necessitating steps to extend the time of polymerisation or prevent it entirely by the addition of organic acids or fatty acids in conjunction with rosin. The influence of adulteration on tung oil was studied by determining the constants of two samples of the oil, which were in turn adulterated with 5%, 10%, and 15% by weight of paraffin oil and many fatty oils. The effects on the sp. gr., refractive index, dispersion, and heat tests are tabulated.

The deodorisation of the oil on a commercial scale is not at present practicable, but some of the difficulties in removing the odour are traceable to the original faulty treatment of the nuts. H. M. LANGTON.

Characteristics of fish and allied oils. A. R. LANGE (Ind. Eng. Chem., 1926, **18**, 9).—The average physical and chemical characters of a season's production of menhaden, Newfoundland cod, herring, whale, and allied oils are tabulated. The author recommends Wijs' method with a saturation period of 1 hr. 15 min. for determining the iodine value. No real success has resulted from attempts to account for the fluctuation in the iodine value of fish oils. Generally oils from fish taken in northern waters yield less stearin than oils from fish from southern waters. H. M. LANGTON.

Rapid determination of iodine value of marine animal oils. B. M. MARGOSCHES, L. FRIEDMANN, and K. FUCHS (Z. deuts. Oel- u. Fett-Ind., 1925, 45, 605—606 ; Chem. Zentr., 1926, I., 791).—The method described previously (cf. B., 1924, 639) can be used for determining the iodine value of marine animal oils, but 10 min. must be allowed for completion of the reaction. If, however, the quantity of iodine added is more than 70% in excess of that required, the reaction is complete within 5 min. A suitable quantity to use is 25 c.c. of 0·2*N*-alcoholic iodine solution per 0·1 g. of oil having an iodine value exceeding 130.

L. A. COLES.

Dugong oil from Australia (Bull. Imp. Inst., 1925, 23, 429—430).—The cream coloured, semi-solid oil obtained from the blubber of the Australian dugong (*Helicore australis*) has the following characteristics: d_{15}^{15} 0·9242, d_{15}^{100} 0·8622, n^{40} 1·458, α^{40} (200 mm. tube) —0·12°, acid value 0·3, saponif. value 204·7, unsaponifiable matter 0·2%, solidif. pt. of fatty acids 34·6°, Reichert–Meissl value 0·3, and Polenske value 1·6. It has a very slight odour and taste free from any fishy character and has been used in Australia as a substitute for cod-liver oil. E. H. SHARPLES.

Effect of ozone on oils. M. V. DOVER and R. B. APPLEBY (Ind. Eng. Chem., 1926, **18**, 63).—Olive oil and two mineral engine oils treated with ozone for varying lengths of time showed increases in sp. gr., acid value, and viscosity, and decrease in iodine value, but little change in refractive index. In the case of olive oil the treatment caused increased interfacial tension, and decreased surface tension and lubricating efficiency, these effects being reversed in the case of mineral oils subjected to the same treatment. H. M. LANGTON.

Mistake in the acid value of crude oils and fats, and use of the "impure acid value." T. HIDAKA (J. Soc. Chem. Ind. Japan, 1925, 28, 1374—1381).—The following definitions are given: "Total acid value" represents the acid value due to free fatty acids and impurities in the oil or fat. "Real acid value" represents the value due to free acids only. "Impure acid value" represents the value due to the impurities other than free fatty acids. 50—100 c.c. of the sample are shaken vigorously with 5—10% of water for from 5 min. to 1 hr., and the resulting emulsion is filtered through a filter paper, yielding a clear oil, the moistened impurities and emulsified fine drops of water being separated. The acid value of this clear oil is the "real acid value," and by subtracting this from the "total acid value" determined on the raw

oil in the usual way, the "impure acid value" is obtained. Percentages of impurities are expressed by $K \times$ "impure acid value." As the value of K, the author obtained 3·9—4 for pressed soya-bean or peanut (arachis) oil, 8·0—9·0 for pressed perilla oil, and 2·0—3·0 for pressed cottonseed oil.

K. KASHIMA.

Formation of the saturated solid *iso*acids during the hydrogenation of fatty oils. I. Presence of *iso*acids in hardened sardine oil. S. UENO (J. Soc. Chem. Ind. Japan, 1925, 28, 1235—1239 ; cf. *ibid.*, 1923, 27, 23).—The formation of *iso*acids during hydrogenation of sardine oil at 160—180° with nickel catalyst was investigated. Solid acids separated from original oil by the lead soap-ether process had a high iodine value. The oil, therefore, probably contained unsaturated *iso*-acids. The solid and liquid acids separated from some samples of the partially hardened oil had similar properties, but the neutralisation value of the former was pronouncedly higher than that of the latter. The hydrogenation takes place in a selective manner, the unsaturated fatty acids of higher molecular weight being more slowly reduced than those of lower molecular weight. In the first stage of the hydrogenation some of the highly unsaturated acids are changed into *iso*acids of the oleic series and then into acids of the saturated series. All samples of the soft hardened oils do not contain highly unsaturated fatty acids, which are completely saturated more readily than acids of the oleic series.

K. KASHIMA.

Distillation of fatty acids. O. HAUSAMANN (Chem.-Ztg., 1926, 50, 41—42).—The factors influencing the distillation of fatty acids are discussed and an air condenser, permitting a lower velocity of the vapours than is necessary in the usual vertical condensers, is suggested. Six condenser tubes are connected so that they form a continuous zig-zag tube having a small uniform slope from the horizontal throughout its length, and fractionation is effected by the insertion of spherical catch pots connected vertically with small cooling coils below. The most advantageous position for the catch pots may be selected by sampling the distillate from test-cocks which can be introduced in intermediate places along the condenser.

E. H. SHARPLES.

Rancidity and yellowing of soaps. G. DE BELSUNCE (Bull. Mat. Grasses, 1925, 191—192).—The author contends that compression into moulds accelerates the production of rancidity whilst the use of very pure fats is a cause also, and in conformity with this latter is the fact that resinous soaps do not become rancid. The use of resinous perfumes hinders or perhaps masks rancidity, and prevents yellowing. When the less pure Leblanc soda was in use rancidity in soap was a rarity. The addition of 1% of sodium hyposulphite, bicarbonate, or carbonate, or ammonia, keeps soap pale for several months and at the same time prevents development of rancidity.

H. M. LANGTON.

Formation of fat in the pig. ELLIS and HANKINS.—See A., Feb., 197.

Colour intensity of oils, etc. FONROBERT and PALLAUF.—See XIII.

Drying of fatty oils. EIBNER.—See XIII.

Solidified tung oil. RASQUIN.—See XIII.

Resin and fatty acids. WOLFF.—See XIII.

PATENTS.

Splitting oils and rendering them odourless, particularly marine animal oils. H. RIEMER (Austr. P. 101,041, 21.3.24).—The oil is heated in an autoclave with water and zinc dust, and mineral acid is added in excess.

L. A. COLES.

Production of soap from waste oils and fats. B. J. A. NYDAM (Dutch P. 13,113, 1.8.21).—Soap manufactured from waste oils and fat is rendered odourless and colourless by passage in aqueous solution over heated plates.

L. A. COLES.

Manufacture of permanent [oil] emulsions. SOC. OF CHEM. IND. IN BASLE (E.P. 245,352, 11.6.25. Addn. to 200,036 ; cf. B., 1923, 863A).—Oils for use in the production of emulsions are halogenated to such an extent that they have the same density as the aqueous solutions with which they are to be emulsified, or, alternatively, they are halogenated to a greater density and subsequently brought to the required density by admixture with material of lower density, soluble in oil, such as paraffin oil or tar-oil. For example, brominated herring oil, d 1·130, mixed with an equal volume of paraffin oil, d 0·900, gives a mixture of d 1·015, which is emulsified with sodium casein solution of the same density.

L. A. COLES.

Apparatus for extraction of oil from blubber and other oil-containing cellular tissues. F. KRUPP GRUSONWERK A.-G. (E.P. 235,550, 28.5.25. Conv., 10.6.24).

Filter for animal fats. (U.S.P. 1,567,989).—See I.

Oxidising oils. (U.S.P. 1,564,331).—See XIII.

Cod liver emulsions (G.P. 419,731).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Mechanism of the formation of lithopone. C. A. MANN (Farbe u. Lack, 1925, 457 ; Chem. Zentr., 1926, I, 781).—On mixing colloidal solutions containing equimolecular amounts of positively charged barium sulphate and negatively charged zinc sulphide a precipitate is obtained which has all the characteristic properties of a true lithopone. The author concludes therefore that the formation of lithopone is due to the mutual saturation of the charges on the individual components which are momentarily produced in the colloidal state.

A. R. POWELL.

Analysis of golden sulphide of antimony. L. HOCK (Kautschuk, 1925, Dec., 11—13).—Golden

sulphide of antimony often contains a higher sulphide than the trisulphide; this is probably the pentasulphide in at least some cases, although tetrasulphide may also be present. After heating in an evacuated tube, antimony sulphide from which soluble sulphur has been removed by extraction with carbon disulphide, the ratio of liberated sulphur to residual black trisulphide indicates the proportion of pentasulphide (or alternatively of tetrasulphide and insoluble sulphur) in the original material. D. F. Twiss.

Preparation of mosaic gold [tin sulphide]. H. Hadert (Chem.-Ztg., 1926, **50**, 7—8).—Mosaic gold (tin sulphide) is best prepared by amalgamating tin turnings (14 kg.) with mercury (6·5 kg.), and heating the powdered amalgam with powdered sulphur (8 kg.) and ammonium chloride (6·8 kg.) at first gently, later more strongly. Adequate regulation of the temperature is of prime importance. By varying the proportions of the starting materials, a diversity of tints ranging from yellow to orange can be obtained. The commercial applications of mosaic gold in the processes of gilding and bronzing are described. L. M. Clark.

Determination of the colour intensity of resins, varnishes, oils, etc. E. Fonrobert and F. Pallauf (Farben-Ztg., 1926, **31**, 967—971).—The authors amplify the "iodine colour value" concept previously described (cf. B., 1925, 931). As a consequence of Wilborn's criticism (B., 1926, 136). it is now stipulated that the sample under test shall be examined in a 10-mm. layer. The colour value may be conveniently measured by dilution of a strong iodine–potassium iodide solution to match the sample, with subsequent titration of the iodine, or alternatively the depth of layer of a suitable iodine solution to match a 10-mm. layer of the sample is noted, and the colour value calculated from the observed proportionality between colour and concentration of iodine solutions. For continuous use an apparatus is described wherein the sample and an adjustable wedge-shaped prism containing iodine solution are viewed from above —in a light-tight box—by means of transmitted light, diffusely reflected. The colour value is read off directly from the graduated prism when in the matching position. For information as to their validity, colour values of the older standards are tabulated. S. S. Woolf.

Drying of fatty oils. A. Eibner (Z. angew. Chem., 1926, **39**, 38—46).—The iodine value does not give a satisfactory measure of the relative value of a drying oil for practical purposes, differences being observed, for example, in the case of isomeric unsaturated fatty acids. During the induction period of the drying of fatty oils, the curve representing the gain in weight is not regular, but is broken by periods of loss in weight occurring regularly between 12 and 2 p.m., *i.e.*, at maximum intensity of illumination. The drying process is more than a simple taking up of oxygen; it is accompanied by a marked and rapid increase in the acid value of the oil which continues until the oil is perfectly dry, after which the value remains constant. For example, the acid value of a sample of linseed oil rose in 60 days from 3·04 to 191·8. Drying oils, when left to dry in a moist atmosphere, absorb moisture, but give it up again subsequently, and the final dry film is in no way impaired thereby. The absorption of water may, however, affect the substratum, *i.e.*, cause rusting of iron or swelling of wood. The drying curve for linseed oil comprises, after the initial increase in weight, a practically straight, horizontal line, but if the oil is dried in bright sunlight the initial gain is followed by a rapid loss and the curve more closely resembles that of poppyseed oil. Molecular weight determinations by Rast's method leave it uncertain whether, in the drying of oils, autoxidation is accompanied by polymerisation. There is also no definite evidence of isomerisation through the influence of light during drying, nor of the formation of hydroxy-acids in the oil film. Peroxide formation, however, does occur, though the peroxides in the final film are present in but small quantity, albeit in a stable form. W. T. K. Braunholtz.

Effect of zinc oxide pigments upon rate of oxidation of linseed oil. F. H. Rhodes and R. A. Mathes (Ind. Eng. Chem., 1926, **18**, 30—31; cf. B., 1924, 916).—Pure refined linseed oil from N. American seed and pigments of known composition were used, the procedure being that previously described (Rhodes and Van Wirt, B. 1923, 1233 A). Pure zinc oxide has no effect on the rate of oxidation of linseed oil, but the presence of lead as impurity may increase the rate of oxidation because of formation of lead driers by interaction of oil and pigment. Presence of soluble zinc compounds causes coagulation at an earlier stage in the oxidation, with formation of a harder and less permeable film. This explains many of the observed phenomena in connexion with the use of zinc pigments, though zinc is not correctly speaking a drier. Addition of zinc sulphate or zinc linoleate is of advantage in the case of lampblack or carbon black paints which often tend to remain sticky for a long period. H. M. Langton.

Solidified tung oil. H. Rasquin (Farben-Ztg., 1926, **31**, 971—972).—Apart from any heat-gelatinised product, tung oil may yield solid bodies by freezing and by exposure to light. The fresh oil has been shown to freeze at +3°, whereas the oils of commerce, having usually experienced long periods of transport through preponderatingly warm regions, are found to remain fluid, albeit viscous, as low as −20°. Incipient polymerisation is suggested as an explanation. Trouble from this source is not common but gentle warming will remedy any congealing or clouding that may occur. The action of light on tung oil converts α-elæostearin, its main constituent, into the solid β-modification which can be isolated by virtue of its relatively sparing solubility in acetone. Its iodine value shows considerable fluctuation, which supports the view that β-elæostearic acid is a conjugated

triply-unsaturated isomeride of linolenic acid. The possibility of utilising this solid product was investigated. Heating with benzol gave only temporary solution with re-precipitation on cooling. Digestion with thickened linseed oil and synthetic resin produced short-oil varnishes of good properties, but the longer-oil varnishes showed a slight tendency to precipitate. S. S. WOOLF.

Determination of resin [and fatty acids in varnishes] by the Wolff–Scholze method. H. WOLFF (Farben-Ztg., 1926, **31**, 917—918).—A repetition of the previously published details of the estimation and separation of fatty and resin acids (cf. B., 1914, 363; 1924, 525). The method comprises saponification and esterification of the separated acids with methyl alcohol and sulphuric acid, alkali-titration of the practically unaffected resin acids as a first approximation, a second esterification of the resin acids to remove any unesterified fatty acids, the former being then weighed and the latter being obtained by difference. The author's experience is that the mean of the volumetric and gravimetric figures gives a satisfactory value for the resin acid content.
S. S. WOOLF.

Receiver for determination of solvent in paints, varnishes, etc. W. TOELDTE (Farben-Ztg., 1926, **31**, 972).—A development of Wolff's receiver (cf. B., 1926, 136) for steam distillation of volatile solvents. A narrow graduated separating-funnel of 50 c.c. capacity and an outlet tube dip into a corked bottle full of water. The solvent collects in the funnel, and the water flows away continuously through the outlet tube. At completion the volume of the solvent is read without the loss involved in running off. It may then be dried and such tests as the sulphuric acid-polymerisation carried out in the same graduated tube. S. S. WOOLF.

See also A., Feb., 210, **Formation of resin in needles of** *Pinus cembra* (PIGULEVSKI and VLADIMIROVA); **Formation of resin in** *Pinus cembra* (PIGULEVSKI). 211, **Formation of resin in** *Abies sibirica* (PIGULEVSKI and GRIGORIEVA).

Colloid chemistry and printing. Treichel.—See VI.

PATENTS.

Production of artificial iron oxide (colcothar, red ochre, etc.) containing no free acids. HEDDERNHEIMER KUPFERWERK U. SÜDDEUTSCHE KABELWERKE, A.-G., Assees. of A. KIRCHNER (G.P. 420,280, 23.4.21).—Iron oxide, free from acid, suitable for use as a pigment, but unsuitable for use in enamelling or in the manufacture of ceramic glazes, is produced by the addition of dry barium carbonate to the oxide at ordinary temperature. Barium sulphate, produced by double decomposition between the carbonate and any sulphuric acid present, produces no deleterious effect upon the colouring properties of the oxide, on account of its insolubility and the relatively small proportion in which it is present. J. S. G. THOMAS.

Manufacturing linoleum. LOBOSITZER A.-G. ZUR ERZEUGUNG VEGETABILISCHER OELE, and G. DUBSKY (E.P. 235,590, 12.6.25. Conv., 16.6.24).—A drying oil is thickened by injecting air until a suitable viscosity is attained, the usual linoleum fillers are incorporated, and the mixture is agitated while under treatment with air or other gas containing oxygen. Improvements in the ease of regulation of the process and the simplicity of the plant necessary are claimed. S. S. WOOLF.

Manufacture of valuable organic compounds. J. Y. JOHNSON, From BADISCHE ANILIN U. SODA FABRIK (E.P. 244,830, 24.9.24).—The oily liquids, chiefly higher alcohols, obtained by catalytic hydrogenation of oxides of carbon as described in E.P. 227,147, 229,714, 237,030, and 238,319 (B., 1925, 189, 338, 784, 900), may be condensed in the presence of zinc chloride, concentrated hydrochloric acid, etc., to yield turpentine substitutes, motor fuels, solvents for cellulose esters, artificial resins, detergents, etc.
S. S. WOOLF.

Purification of [crude] abietic acid and production of pure abietates or abietic acid. G. H. DUPONT (E.P. 244,980, 24.4.25).—Crude abietic acid (colophony) is suspended in alcohol and sufficient alkali added to neutralise one-quarter of the acid. An acid salt is precipitated which may be separated and further saponified, yielding pure rosin soaps, or decomposed by strong acid to regenerate pure abietic acid. S. S. WOOLF.

Oxidising oils. A. EISENSTEIN, Assr. to G. SCHICHT, A.-G. (U.S.P. 1,564,331, 8.12.25. Appl., 18.1.21).—Linseed oil or other unsaturated oil is mixed with molten paraffin wax and the congealed mixture powdered and exposed to the action of air at ordinary temperature. The dispersed linseed oil is rapidly oxidised. The wax is subsequently melted away from the solid oxidation product. Alternatively the oil may be dispersed by the aid of common salt and oxidised at 100°, the salt being subsequently removed with water. T. S. WHEELER.

Film-forming composition. H. A. GARDNER (U.S.P. 1,564,664, 8.12.25. Appl., 7.4.25).—A resin obtained from *p*-toluenesulphonamide and an aldehyde is dissolved with nitrocellulose or cellulose acetate in a lacquer solvent, such as butyl acetate.
T. S. WHEELER.

Production of phenol-methylal resin. C. B. CARTER and A. E. COXE, Assrs. to S. KARPEN AND BROS. (U.S.P. 1,566,817, 22.12.25. Appl., 23.8.22).—Methylals condense with phenols or cresols in presence of water and an acid at 180° under pressure to form soluble resins similar to those obtained from phenol and formaldehyde under similar conditions.
T. S. WHEELER.

Producing a phenolic condensation product. C. B. CARTER, Assr. to S. KARPEN AND BROS. (U.S.P. 1,566,823, 22.12.25. Appl., 26.7.24).—The aqueous solution of ammonium chloride and hexamethylenetetramine obtained by heating methylene chloride

and ammonia together in aqueous solution is boiled under reflux with phenol. A fusible resin of the usual phenol–hexamethylenetetramine type is produced.
T. S. Wheeler.

Paint and varnish remover. E. R. Littmann, Assr. to Commercial Solvents Corp. (U.S.P. 1,568,753, 5.1.26. Appl., 13.3.25).—The composition claimed comprises a saturated aqueous solution of sodium nitrophthalate, acetone, ethyl alcohol, benzol, and toluol.
B. M. Venables.

[Resinous] substances for the manufacture of optical apparatus. Badische Anilin u. Soda-Fabr., Assees. of H. Ramstetter (G.P. 416,753, 31.1.23).—Transparent condensation products of carbamide and its derivatives with substances capable of yielding formaldehyde are used as a substitute for quartz glass; they absorb ultraviolet rays but slightly.
A. J. Hall.

Preparation of oily and resinous condensation products from aromatic hydrocarbons and aralkyl halides. Farbw. vorm. Meister, Lucius, u. Brüning, Assees. of F. Marschall (G.P. 417,442, 4.7.20. Addn. to 416,904; cf. B., 1926, 100).—The products are obtained by heating mixtures of aralkyl halides and polynuclear aromatic hydrocarbons, or their halogen derivatives, in the presence of hydrogen chloride. For example, on heating a mixture of naphthalene and a solution of hydrogen chloride in benzyl chloride, interaction proceeds vigorously at 90—100° with evolution of hydrogen chloride, and is completed by further heating to 130° for 1 hr. After removing uncondensed constituents by treatment with a current of steam, a pale, viscous resinous product remains.
L. A. Coles.

Manufacture of a preservative. F. Trachsel (G.P. 419,097, 7.12.24).—A solution of an alkali silicate is mixed with oil of turpentine, the mixture allowed to settle, and the excess of turpentine removed. The method of mixing may be such that a maximum quantity of air is incorporated. Material impregnated with the mixture may be subsequently treated with a suitable reagent to cause precipitation of silicic acid, magnesium silicate, or aluminium silicate.
S. Binning.

Manufacture of resinous condensation products from phenols and sulphur. Ges. für Chem. Ind. in Basel (Swiss P. 105,855–8, 2.12.22. Addn. to Swiss P. 92,408; cf. E.P. 186,107, B., 1922, 905 a).—Homologues or substituted derivatives of phenol, *e.g.* β-naphthol, pyrocatechol, resorcinol, and pyrogallol, are heated with sulphur in the presence of a basic catalyst, and thereby converted into artificial resins. An increase in the proportion of sulphur used results in resins having a higher m.p., greater hardness, and decreased solubility in the usual solvents. The resins may be "filled" with large quantities of other resinous substances obtained from sulphur and amines or phenols and for this purpose the amines or phenols may be added to the reaction mixture immediately before conversion into resin by the process described above. The solubility of the resins is decreased by again heating them, with or without pressure, together with further quantities of sulphur or with substances containing active methylene groups such as hexamethylenetetramine and resins containing such groups. The sulphur-containing resins obtained from β-naphthol are brownish yellow, and insoluble in caustic alkalis, acetone, ether, alcohol, and benzene; from pyrocatechol, brown, slightly soluble in cold and easily soluble in hot water, very easily soluble in aqueous solutions of sodium carbonate or caustic alkalis, and insoluble in alcohol, acetone, and benzene; from resorcinol, insoluble in water, benzene, and toluene, and soluble in alcohol, acetone, ether, and aqueous solutions of sodium carbonate and caustic soda; from pyrogallol, brownish-black, easily soluble in water, ether, alcohol, and alkalis, and insoluble in benzene and toluene.
A. J. Hall.

Resin substitute produced by Friedel–Crafts reaction. Chem. Fabr. auf Aktien vorm. E. Schering, Assees. of E. Freund and H. Jordan (U.S.P. 1,570,469, 19.1.26. Appl., 25.8.23).—See E.P. 203,997; B., 1925, 46.

Finely divided solids (G.P. 419,758).—See I.

Preventing discoloration of canned foods (U.S.P. 1,567,163).—See XIX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Formic acid as a coagulant for *Hevea* latex. O. de Vries, W. Spoon, and R. Riebl (Comm. Central Rubber Stat. Buitenzorg, 1925, **50**, 32 pp.; Arch. Rubbercultuur, 1925, **9**, [9]).—Coagulation of *Hevea* latex with formic acid gives rubber with a rate of cure slightly below the average, but within the limits of ordinary variation; the rubber also is a little more plastic and its keeping properties are as good as those of rubber coagulated with acetic acid. The differences generally are so small as to be negligible.
D. F. Twiss.

Matured rubber. Influence of different factors in preparation. Loss in weight. Inner properties. Large-scale preparation of "slab" rubber. O. de Vries and W. Spoon (Comm. Central Rubber Stat. Buitenzorg, 1925, **52**, 60 pp.; Arch. Rubbercultuur, 1925, **9** [11]).—Variation in the maturation period of the coagulum from 2 to 10 days gave no difference in the inner properties of the final rubber, which also were not affected by removal of the slimy layer by washing and by treatment of the slabs with antiseptics such as β-naphthol. The maturing of slab from latex drawn from trees which had been rested effected an increase in the rate of vulcanisation, but the rate was lower than for ordinary matured rubber. A test over 3 days showed maturation to result in an average loss in yield of 1·26%. The degree of uniformity in the rate of vulcanisation of matured rubber is much less than for ordinary smoked sheet rubber. The rate of cure of "Amber blanket" rubber prepared from native "slab" (*i.e.*, matured)

rubber is influenced by the use of alum as coagulant which reduces the rate to approximately that for ordinary rubber. Although large quantities of matured rubber have been prepared to the order of certain manufacturers, the likelihood of adoption of this form of rubber as a standard market product seems remote. D. F. Twiss.

Plasticity determinations on crude rubber. IV. Difference between smoked sheet and pale crêpe. O. de Vries (Comm. Central Rubber Stat. Buitenzorg, 1925, **51**, 31 pp.; Arch. Rubbercultuur, 1925, 9 [9]).—The difference in the factors in the preparation of crêpe and smoked sheet rubber has little influence on the relative plasticity of the two forms. Crêpeing has a distinct plasticising effect relative to sheeting, but soaking in water and heating or smoking affect the plasticity of the fresh rubber to a remarkably slight extent. The earlier results indicating the greater hardness of crêpe rubber are attributed to the effect of alteration by storage. D. F. Twiss.

Relative rate of oxidation of monophase caoutchouc gel and of raw rubber. H. Feuchter (Kautschuk, 1925, Aug., 11—12; Chem. Zentr., 1925, ii, 2299).—Rubber purified by diffusion (cf. B., 1925, 557) absorbs oxygen less rapidly than ordinary rubber but the accompanying physical deterioration is much greater. D. F. Twiss.

Vulcanised latex and its commercial applications. P. Schidrowitz (Trans. Inst. Rubber Ind., 1925, **1**, 270—278).—A review of the subject. Materials prepared from vulcanised latex are found to maintain their condition better than the corresponding commercial products prepared in the ordinary way. D. F. Twiss.

Tensile strength of cold-vulcanised rubber. A. Zeitlin (Trans. Inst. Rubber Ind., 1925, **1**, 255—260).—When vulcanising thin layers of rubber by a solution of sulphur chloride, increase in the period of immersion or in the concentration of the solution has an effect analogous to that of increase in the time, or of the proportion of sulphur, in the hot vulcanisation process. The cold-vulcanisation products yield a similar stress-strain curve to ordinary vulcanised rubber, the "slope" being comparable and the tensile strength ranging up to 2550 lb. per sq. in. D. F. Twiss.

Rate of combination of sulphur with rubber in hard rubber [vulcanite]. W. E. Glancy, D. D. Wright, and K. H. Oon (Ind. Eng. Chem., 1926, **18**, 73—75).—On progressive vulcanisation a mixture of rubber (70) and sulphur (30) attains the typical character of vulcanite when the combined sulphur is approximately 23·5% (coefficient of vulcanisation approx. 32) which would correspond with a compound $C_{10}H_{16}S$. Vulcanisation beyond this stage effects very little increase in tensile strength and the product is still flexible. The volume of the product is less than that of its ingredients; assuming the specific gravity of sulphur to remain constant, that of rubber in vulcanite must be near 0·998. The addition of an organic accelerator to the above mixture causes an increase in the rate of vulcanisation, but does not increase the strength of the product; with hexamethylenetetramine indeed, this appears to be influenced adversely. D. F. Twiss.

Elasticity-hysteresis and its significance for the colloid structure of rubber. H. Feuchter (Kautschuk, 1925, Dec., 6—11).—If thin strips of *Hevea* rubber are warmed, then stretched to 1500%, and cooled in this state, they retain most of the stretch when the force is removed; when heated with water the rubber again retracts and the curve showing the relation between the extent of recovery and the temperature (between 20 and 95°) follows an S-shaped course somewhat similar to that of the ordinary stress-strain curve. Thin strips of raw rubber can be stretched much further and to a greater degree of tension by a sudden application of force than by a gradual pull. The changes involved in the elastic phenomena exhibited by rubber are colloidal reactions and neither purely physical nor purely chemical in character; the physical or chemical conception of a molecule does not hold for rubber. The elasticity of rubber is a thermodynamic function of its structure and its elastic behaviour involves correlated dynamic, thermal, chemical, optical, and electrical changes. (Cf. B., 1926, 100.) D. F. Twiss.

Gas black for rubber manufacture: effects and detection of grit. G. Gallie (Trans. Inst. Rubber Ind., 1925, **1**, 247—254).—Gas black, a common reinforcing agent for rubber, is frequently contaminated by the presence of carbonaceous gritty particles which are unable to pass even an "80" sieve. These coarse particles may survive the mechanical operations involved in forming a mixture with rubber and adversely affect the tensile strength of the product. D. F. Twiss.

Caoutchouc as a dispersion medium. H. Pohle (Kolloid-Z., 1926, **38**, 75—76).—The irregularity with which, in practice, fillers are dispersed in rubber is discussed. A prominent contributory cause is the tendency for very fine powders to "pack" to form secondary particles which are often exceedingly resistant to disintegration. Measurement of the light absorption of thin films of rubber-filler mixtures gives useful information about the degree of dispersion of the latter, and the progress of the mixing process. N. H. Hartshorne.

Vulcanisation in hot air. B. Bysow (India-Rubber J., 1926, **71**, 99—100).—See B., 1926, 101.

Application of micrurgy to colloid chemistry. Hauser.—See I.

Analysis of golden sulphide of antimony. Hock.—See XIII.

Patents.

Concentrating or coagulating rubber emulsions. Anode Rubber Co. From P. Klein

and A. Szegvari (E.P. 245,177, 31.7.24).—In the electrophoretic deposition of rubber on an anode or on a diaphragm surrounding the anode, there are inserted between the anode and cathode, partitions which may be impermeable, semi-permeable, or permeable to liquids and fixed or movable according to needs. Amongst the advantages are the possibility of regulating the thickness of the deposit over the anodic surface and protection of the deposit from cathodic gases. D. F. Twiss.

Manufacture of rubber products containing cork. R. Grimoin-Sanson (F.P. 590,871, 22.2.24).—Powdered cork, after prolonged digestion with 5% sodium carbonate solution, is dried, impregnated with 10% rubber solution, and mixed with a stronger rubber solution. After addition of sulphur, the mass is moulded into shape and vulcanised, yielding a light, uniform product suitable for the manufacture, *e.g.*, of tyres and electrical insulators. L. A. Coles.

Rubber cement. B. P. Taylor, Assr. to Taylorall Inc. (U.S.P. 1,566,566, 22.12.25. Appl., 5.4.24).—A rubber cement consists of rubber latex, water, and furfuraldehyde, with addition of zinc oxide, sulphur, chloronaphthalene, ethyl phthalate, etc. T. S. Wheeler.

Chemical process for etching rubber for printing blocks. Soc. d'Exploit. des Proc. d'Impression Sardou (E.P. 241,542, 7.10.25. Conv., 15.10.24).

Compound for accelerating the vulcanisation of caoutchouc. V. Lefebure and A. J. Hailwood, Assrs. to Brit. Dyestuffs Corp. (U.S.P. 1,570,752, 26.1.26. Appl., 1.10.23).—See E.P. 218,854; B., 1924, 757.

XV.—LEATHER; GLUE.

Methods for measuring the enzyme activities of bating materials. J. A. Wilson and H. B. Merrill (J. Amer. Leather Chem. Assoc., 1926, 21, 2—18).—The enzyme activities of bating materials are measured by the amounts of various substrates hydrolysed by a given amount of the material under certain conditions. Keratose solution, containing 2 g. of keratose, is pipetted into a 1-litre flask, 100 c.c. of a disodium phosphate—sodium citrate—boric acid buffer solution of p_H 8·0 are added, the mixture is diluted to 950 c.c., and warmed to 40°. Sufficient bating material should be weighed out to digest 40% of the keratose in 1—3 hrs., mixed with 25 c.c. of water at 40°, added to the flask, and the mixture made up to 1000 c.c. The flask is placed in a thermostat at 40° and the unchanged keratose determined in aliquot portions of the mixture after definite intervals of time. From the figures obtained, a graph is plotted and the time determined after which 40% of the keratose has been dissolved. The keratose-digesting power of the bating material is the reciprocal of the time in hours required for 1 g. of the enzyme to digest 40% of 2 g. of keratose dissolved in 1 litre of aqueous solution at 40° and p_H 8·0. The elastin-digesting power of an enzyme is the reciprocal of the number of grams of the bating material per litre required to remove all the elastin fibres from calfskin in 24 hrs. at 40° and p_H 7·9. Pieces of limed, unhaired and washed calfskin are placed in a series of bottles containing a phosphoric acid-sodium hydroxide buffer solution at p_H 7·9, increasing weighed quantities of the bating material are added to the respective bottles, which are kept at 40° for 24 hrs. The calfskin pieces are washed, dehydrated with alcohol, sectioned, stained with Weigert's stain, mounted, and examined microscopically to determine the lowest concentration of bating material which has removed all the elastin. The collagen-digesting power of an enzyme is the reciprocal of the concentration of enzyme in g. per litre required for the digestion of 20% of 5 g. of purified hide powder in 1 litre of aqueous solution at 40° and p_H 7·9 in 3 hrs. Increasing amounts of enzyme preparation are weighed into various bottles, each containing hide powder equivalent to 0·5 g. of collagen, 10 c.c. of a buffer solution of p_H 7·9, and 90 c.c. of water. A control test is also made. The bottles are kept at 40° for 3 hrs., the nitrogen determined in the solution, and the dissolved collagen calculated. The results are plotted and the concentration noted at which 20% of the collagen is dissolved. The power of a bating material to digest casein bears no relation to its keratose-, elastin-, or collagen-digesting power. The methods of Fuld-Gross and of Northrop (J. Gen. Physiol., 1922, **5**, 263) can be used to determine the lowest concentration of bating material which will digest all the casein in 1 hr. The lipolytic activity of the bating material can be represented by the fraction $1/g.$, where $g.$ is the number of grams of material required to hydrolyse 24% of a 2·5-g. sample of olive oil in 1 hr. in presence of 12 c.c. of a buffer solution containing 670 c.c. of 0·1N-ammonium chloride, 330 c.c. of 0·1N-ammonia, and 200 c.c. of water. D. Woodroffe.

Tannin content of Western hemlock after immersion in sea water. H. L. Templeton and E. C. Sherrard (Ind. Eng. Chem., 1926, **18**, 101—102).—Samples of hemlock bark, untreated and after immersion in sea water for 30, 60, and 90 days respectively, were analysed for tannin content and sodium chloride. Immersion in salt water for periods of 90 days or less had no observable effect on the tannin content or the non-tannin content. The total and soluble solids were slightly greater and the insoluble matter increased. There was no relation between the salt content and the tannin content. The colour was darkened by the immersion.
D. Woodroffe.

Effect of vegetable tanning upon the combination of collagen with acid. J. A. Wilson and A. W. Bear (Ind. Eng. Chem., 1926, **18**, 84).—According to the Procter-Wilson theory of vegetable tanning (J.C.S., 1916, **109**, 1327) the capacity of collagen to combine with hydrogen ions should decrease as the amount of combined tannin increases. This deduction has been proved correct. Samples of hide powder were tanned in oak bark extract liquors for different lengths of time, the tanned

powders washed in running water to remove uncombined tan and free acid, dried, and analysed. When samples of each powder were treated with $0 \cdot 01N$-sulphuric acid, the p_H value of the residual liquor was found to increase as the amount of combined tannin in the powder increased.

D. WOODROFFE.

Acidity of chrome-tanned leather. A. C. ORTHMANN (J. Amer. Leather Chem. Assoc., 1926, **21**, 30—33).—Pickled calfskins containing 4·5% of sulphuric acid (dried skin) were tanned with 35% of a chrome tanning liquor of 33% basicity (Schorlemmer) prepared by reduction with maize sugar. After drumming for 5 hrs. 1% of sodium bicarbonate was added in 2 portions, the hides were "horsed up" for 24 hrs., rinsed, neutralised with 1% sodium bicarbonate, washed, dyed, fat-liquored with soap and oil, dried, and finished. Samples of the leather were taken at various stages during the process and analysed. No hydrolysable acid was present after the second treatment with sodium bicarbonate. Leather is entirely free from hydrolysable acid if proper care is exercised in neutralising and washing.

D. WOODROFFE.

Destructive action of sulphuric and hydrochloric acids upon leathers. J. A. WILSON (Ind. Eng. Chem., 1926, **18**, 47—50).—Alternate strips from a sample of chrome-tanned leather were tested for tensile strength and the others were wet with solutions of different p_H value ranging from $0 \cdot 5N$-sodium carbonate to $5N$-sulphuric acid, blotted, air-dried, kept for 6 months, the tensile strength was determined, and the broken pieces were analysed for water and sulphuric acid. Up to an acid content of 10%, the leather treated with acid had the same tensile strength as the untreated pieces, but the strength was impaired with a greater amount of acid, and with more than 15% of sulphuric acid the leather was rapidly destroyed. Comparative tests showed that chrome leather combined with sulphuric acid more readily than did vegetable-tanned leather and it was more easily destroyed by free sulphuric acid. The retannage of chrome leather with vegetable tanning materials increased its resistance to the destructive action of sulphuric acid, and it was possible to prepare thin sheets of leather by coating the surface of chrome leather with vegetable tannin and then soaking it in $6N$-sulphuric acid, which dissolved all the leather except the tannin-coated film. Hydrochloric acid was less harmful than sulphuric acid owing to its volatility, but when volatilisation was prevented, chrome leather was destroyed more quickly by hydrochloric than by sulphuric acid in solutions stronger than $3N$. Tests on pieces of vegetable-tanned calf butt 13 and 20 years old and containing 0·6% and 0·53% of sulphuric acid respectively, showed that the tensile strength had not decreased. The maximum acid content for vegetable-tanned leather seems to be from 0·6% to 4·0%.

D. WOODROFFE.

Dispersion of powdered egg yolk. R. M. COBB and F. S. HUNT (J. Amer. Leather Chem. Assoc., 1926, **21**, 18—22).—The lowered emulsifying power of old egg yolk is probably due to the acids split off from the lecithin molecule. Attempts have been made to restore the emulsifying properties by mixing dried egg yolk with alkali and then incorporating as much water as the mixture would hold without becoming liquid. The weight of water taken up was small until the p_H reached 8·0. At p_H 8·6 the emulsion was as good as that obtained with liquid egg yolk. The slightest trace of acid, however, caused clotting.

D. WOODROFFE.

Determination of water content of liquid glue. W. A. KINGMAN (Ind. Eng. Chem., 1926, **18**, 93—94).—The glue is mixed with tetrachloroethane (50 c.c. to 15 g. of glue) and the mixture distilled into a burette-condenser through a side arm attached to the burette. The burette is prolonged upwards to a condenser, and at the bottom, above the stopcock, are placed 3 c.c. of tetrachloroethane. The condensed water collects above this, and since the b.p. of tetrachloroethane is higher than that of water all the water vapour is carried over. Any drops of water are washed down from the walls of the burette by naphtha, and the volume of water is read off in the burette.

D. G. HEWER.

Effect of light on tannin formation. MICHEL-DURAND.—See A., Feb., 208.

Cause of "gulf" disease. BIGINELLI.—See XXIII.

PATENTS.

Manufacture of tanning substances. O. RÖHM (U.S.P. 1,569,578, 12.1.26. Appl., 5.3.24).—A tanning material is prepared by mixing together an iron salt, an alkali silicate, and sulphite-cellulose waste liquor.

L. A. COLES.

Process for removing acids from leather, especially from chrome leather. R. HITSCHMANN (Austr. P. 99,929, 29.3.24).—The hides after tanning are plunged in water which may contain an electrolyte. An electric current, preferably a direct current, is passed through the water, the electrodes being in the front of the bath and separated by a diaphragm from the rest of the bath. Not only acid but also calcium compounds are removed from the leather.

S. BINNING.

Treatment of hides and skins for manufacture into leather. D. MCCANDLISH and W. R. ATKIN (U.S.P. 1,570,383, 19.1.26. Appl., 22.7.25).—See E.P. 235,678; B., 1925, 683.

Solidifying liquid colloids (G.P. 420,637).—See I.

XVI.—AGRICULTURE.

Base exchange in relation to alkali soils. W. P. KELLEY and S. M. BROWN (Soil Sci., 1925, **20**, 477—495).—The nature of the exchangeable bases in a number of alkali soils has been studied by leaching samples with N-ammonium chloride solution and determining the bases in the filtrate, due

allowance being made for the water-soluble bases and the carbonates present. The results emphasise the important changes in the chemical and physical properties of the soils caused by reaction with alkali salts. In certain cases, sodium has taken the place of most of the replaceable calcium and magnesium. The permeability of such soils is markedly diminished when the soluble salts are leached out and they cannot be dealt with simply by flooding and drainage. On the other hand, some soils containing large amounts of soluble sodium salts also show relatively high concentrations of calcium and magnesium and do not contain abnormal amounts of replaceable sodium. This type may be reclaimed by flooding and drainage alone. The insoluble but exchangeable components may play a part as important as the soluble salts in determining the toxicity of alkali soils.

C. T. GIMINGHAM.

Alkali [soil] studies. IV. Tolerance of oats for alkali [salts] in Idaho soil. R. E. NEIDIG and H. P. MAGNUSON (Soil Sci., 1925, **20**, 425—441).—Four successive crops of oats were grown in soil to which varied amounts of sodium chloride, carbonate, and sulphate were applied, singly and in combination. In general, the results were similar to those obtained with barley (cf. B., 1926, **24**). The toxic concentration of sodium carbonate was about 0·4% and of sodium chloride about 0·25%; sodium sulphate was not toxic up to 1·5%, except to the first crop.

C. T. GIMINGHAM.

Relationship between soluble iron and colloids in certain residual clays. G. R. MACCARTHY (Soil Sci., 1925, **20**, 473—475).—In the case of greatly weathered clays and clayey materials, the ratio of the amount of iron soluble in dilute hydrochloric acid per g. of the material to the amount per g. of the colloid fraction of the material gives a figure for the percentage of colloidal matter present which agrees quite closely with that obtained by the water adsorption ratio (cf. Gile and others; A., 1924, ii, 796). With clays containing much unweathered material, and, therefore, much iron not in a colloid condition, the results given by the two methods are quite discordant. It is suggested that determination of soluble iron may prove a simple means of estimating the degree of weathering, or, if the material is known to be completely weathered, of determining the percentage of colloidal material.

C. T. GIMINGHAM.

Movable lysimeter for soil studies. F. L. DULEY (Soil Sci., 1925, **20**, 465—471).—The lysimeter consists essentially of a large galvanised iron cylinder with a removable bottom provided with a drainage hole. It is fitted over a core of soil trimmed to the proper size, the space between the cylinder and soil being then filled with high-grade asphalt which serves to hold the mass of soil in place. The soil is then cut away at the base, the bottom of the cylinder fixed, and the whole apparatus, containing a large block of undisturbed soil, can be removed by means of suitable tackle to a permanent concrete pit prepared for it.

C. T. GIMINGHAM.

Soil acidity. GEHRING (Landw. Vers.-Stat., 1925, **104**, 164—177).—Many clay and heavy loam soils which give increased yields of crops on liming show little or no "exchange" acidity. With such soils there is a parallelism between the response to liming and the degree of saturation for calcium, *i.e.*, the relation between the percentage of exchangeable calcium and the total percentage of calcium which the soil will absorb. When the exchangeable calcium is 70% or more of the total which the soil will take up, no response from liming is to be expected. The application of these results to soils of other types is discussed.

C. T. GIMINGHAM.

Vegetation experiments on soil acidity. MITSCHERLICH (Landw. Vers.-Stat., 1925, **104**, 158—164).—The reaction of some soils, as judged by laboratory tests, may vary greatly under different conditions. To get over this and other difficulties, a method, involving vegetation experiments, is proposed in order to provide a basis for practical recommendations as to liming and suitable manuring of individual soils. The effect of heavy applications to the soil of physiologically acid and alkaline mixtures of manures on both an acid-sensitive plant (mustard) and an alkali-sensitive plant (oats) is investigated. The results of such experiments with 50 soils and the conclusions to be drawn from them are tabulat d.

C. T. GIMINGHAM.

Effect of calcium carbonate, gypsum, and sodium carbonate on soils rendered acid with hydrochloric and sulphuric acids. F. MÜNTER (Landw. Vers.-Stat., 1925, **104**, 177—182). **Vegetation and field experiments on soils showing "exchange" acidity.** RÖSSLER (*Ibid.*, 182—202). **Effect of plants on soil reaction and its importance in vegetation experiments.** KRÜGER (*Ibid.*, 202—215).—These three papers deal with different aspects of the subject of soil acidity with particular reference to the bearing of "exchange" acidity on the varied effects obtained by liming different types of acid soils. The originals should be consulted for details.

C. T. GIMINGHAM.

Potassium xanthate as a soil fumigant. E. R. DE ONG (Ind. Eng. Chem., 1926, **18**, 52—55).—Potassium and sodium xanthates are soluble in water, stable in the air, safe to manipulate, capable of controlled effects, and effective nematode poisons. Their effect depends upon their decomposition, first to xanthic acid, then to alcohol and carbon disulphide (which acts as the fumigant), and the decomposition may be made partly rapid and partly slow, *e.g.*, by addition of superphosphate and sulphur, the former causing rapid decomposition of part of the xanthate, followed by a more gradual decomposition of the remainder under the influence of the sulphur. Potassium xanthate has a retarding effect on the growth of seedlings, but the duration of the retarding action can be shortened by the

addition of a neutralising agent in quantity sufficient to cause xanthic acid to be liberated immediately. No injurious effects have been noted on citrus or dormant deciduous trees. D. G. HEWER.

Rate of oxidation [in soil] of different forms of elementary sulphur. R. H. SIMON and C. J. SCHOLLENBERGER (Soil Sci., 1925, **20**, 443—449).—Samples of four commercial forms of sulphur were mixed with soil and incubated under controlled conditions. The rate of oxidation, as measured by determinations of sulphates at definite intervals, was dependent on the fineness of the material; and the most complete oxidation was correlated with the greatest increase of hydrogen-ion concentration. Determination of the residual sulphur by the acetone extraction method (cf. B., 1926, 70) was used as a check. C. T. GIMINGHAM.

Effect of sulphur upon nitrogen content of legumes. J. R. NELLER (Ind. Eng. Chem., 1926, **18**, 72—73).—Sulphur after mixing with a warm arable soil undergoes a comparatively rapid oxidation to trioxide, and legume crops showed an increase in their nitrogen content when grown in such soil. In the case of lucerne this increase varied from 32·7 to 44·2%. The sulphur content of the plants was also increased, but there was little effect upon the intake of other plant foods. Non-leguminous crops do not appear to be influenced in any way by the sulphur. D. G. HEWER.

Relative toxicity of the arsenates of calcium. S. B. HENDRICKS, A. M. BACOT, and H. C. YOUNG (Ind. Eng. Chem., 1926, **18**, 50—51).—Experiments on the toxicity of all the known compounds of the system $CaO—As_2O_5—H_2O$ to locusts and boll weevils indicate that the acid arsenates are more toxic than the basic, probably because basic arsenates must be partially hydrolysed to compounds giving more "soluble" arsenic before toxic results are produced. Probably with all gross-feeding insects toxicity decreases with increase in the molecular ratio $CaO : As_2O_5$ in combination and is not affected by $Ca(OH)_2$ in the presence of $Ca_3(AsO_4)_2,H_2O$. The ratio of As_2O_5 in the body to that in the fæces of the insect, and the average longevity, were found to be satisfactory criteria of toxicity for arsenicals. D. G. HEWER.

[Seed] stimulation. TACKE (Landw. Vers.-Stat., 1925, **104**, 153—158).—A review of results obtained at six experiment stations in Germany. It has been claimed that preliminary soaking of seeds in solutions of salts of magnesium, manganese, and other metals, or of mixtures of various substances stimulates germination and increases yields. The experiments discussed include trials with seeds of a number of crops and in no case was any significant advantage obtained by any of the treatments. C. T. GIMINGHAM.

See also A., Feb., 139, **Determination of hydrogen-ion concentration of [soil] suspensions by the quinhydrone method** (HETTERSCHIJ and HUDIG). 208, **Effect of light on tannin formation** (MICHEL-DURAND); **Effect of light on alkaloid content of *Lupinus luteus*** (SABALITSCHKA and JUNGERMANN). 210, **Nitrogen constituents of celery plants in health and disease** (COONS and KLOTZ).

Lead arsenates. CAMBI and BOZZA.—See VII.

PATENTS.

Production of finely crystalline urea. WARGÖNS AKTIEBOLAGET, and J. H. LIDHOLM (F.P. 586,025, 17.9.24. Swiss P. 110,748, 12.9.24. Conv., 29.10.23).—Urea solutions concentrated to such an extent that they set to a solid mass on cooling, *i.e.*, containing not more than 10% of water, are either cast into blocks or allowed to fall as drops into a gas at such a temperature that the drops solidify as they fall. The product, whether in the form of blocks or granulated, remains dry on storing, and after granulation, can easily be applied to the soil by the usual distributing machines. L. A. COLES.

Apparatus for the electrometric determination of the hydrogen-ion concentration of soils, waters, chemical solutions, etc. M. TRÉNEL (G.P. 399,410, 7.12.23, and 400,273, 6.1.24).—The solution to be tested is connected through a porous diaphragm with the comparison solution, and insoluble electrodes saturated with hydrogen are dipped into each liquid. The diaphragm is previously saturated with a solution of potassium chloride or with a jelly saturated with potassium chloride. The whole apparatus has the appearance of a Bunsen cell and, when packed in a suitable box, is portable and may be used for field work. A. R. POWELL.

Insecticides. W. J. MELLERSH-JACKSON. From CALIFORNIA SPRAY-CHEMICAL CO. (E.P. 245,179, 18.8.24).—Specially purified mineral oils, having insecticidal but not phytocidal properties (designated "phytonomic" oils), are obtained by distillation, sulphonation, and filtration of petroleum oils, and, for use as insecticides, are combined with a relatively volatile petroleum distillate (kerosene), to which may be added an emulsifying agent (sulphite-pulp waste liquor or dextrin) and water. The phytocidal properties of petroleum oils are stated to be due mainly to unsaturated hydrocarbons, sulphur, the lighter cyclic hydrocarbons, and organic acids. Phytonomic oil is colourless, transparent, has practically no petroleum taste or odour, and is practically non-volatile and non-drying at ordinary temperature, d 0·8917—0·8092, viscosity (Saybolt) 50—350 sec. at 38°; it is coloured only a light straw yellow by concentrated sulphuric acid, contains only about 0·01% S, and is either viscous or solid. Suitable oils are already marketed, but for different purposes, *e.g.*, "Wyrol," "Oronite Crystal Oil," "Nujol," "Marcol." C. O. HARVEY.

Fungicide and insecticide. O. F. HEDENBURG, Assr. to F. O. MOBURG (U.S.P. 1,568,455, 5.1.26. Appl., 14.2.21).—A composition which readily forms a suspension in water, comprises Bordeaux mixture together with dextrin, casein, and lead arsenate and/or a soluble chromate. C. O. HARVEY.

Material for combating pests. H. Weil (G.P. 419,324, 9.8.23).—Dichlorodiphenylmethane, used either alone or mixed with other substances, is claimed as a material for destroying pests in all stages. C. T. Gimingham.

Material for combating plant and animal pests. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of K. Görnitz and H. Goebel (G.P. 419,390, 18.5.24).—Diricinoleicdisulphonic acid or its salts ("Turkon oil") are added to spray fluids such as Bordeaux mixture, arsenic mixtures, barium chloride, and polysulphides, to increase wetting and adhesive properties. They possess an advantage over Turkey-red oil since metal soaps or other reaction products are not formed with salts of alkaline earths or heavy metals; hard water can be used without the formation of flocculent calcium soaps. The material is used at the rate of about 1%. C. T. Gimingham.

Mixtures containing finely divided copper compounds for combating plant pests. Farbw. vorm. Meister, Lucius, u. Brüning (G.P. 419,460, 21.5.21. Addn. to 416,899; cf. B., 1926, 103).—The products of sulphonation of such substances as naphthol- and anthracene-pitch, synthetic resins, colophony and other natural resins have an advantage over the salts of aromatic sulphonic acids since they may be added to liquors containing copper compounds in appreciably smaller proportion and give a more finely divided precipitate. The mixtures may be incorporated with other fungicides and insecticides and also find uses as disinfectants and in the dyeing industry. C. T. Gimingham.

Material for combating plant pests. Farbw. vorm. Meister, Lucius, u. Brüning (G.P. 419,461, 5.7.24).—The material consists of the iron oxide (Fe_3O_4) sludge obtained in the reduction of nitro-compounds. It is used alone, or, for example, with sulphur or copper or arsenic compounds or other insecticides and fungicides; or it is dried and the fine powder used with wetting and spreading materials. Mixtures of arsenates or arsenites with the iron sludge are harmless to plants, since any free acid is neutralised by the iron oxide. The sludge may also be used as the basis of weed-killing preparations; for example, mixed with ferrous sulphate solution, or as a dust, by treatment with sulphuric or arsenic acid and reduction of the product to a fine dry powder. C. T. Gimingham.

Insecticide for plants. S. Kreidl (Austr. P. 100,216–7, 30.8.23).—(A) An insecticide for spraying plants consists of a 1—2% or 5—10% emulsion of hydrogenated phenols, *e.g.*, hexalin or methylhexalin, either alone or in admixture with other substances. (B) The insecticide consists of an emulsion of a solution in pyridine or quinoline bases of hydrogenated aromatic hydrocarbons, such as tetralin or decalin, or of phenols, such as hexalin or methylhexalin. The emulsions are stable and permanent and the addition of soap as a stabiliser is unnecessary. A. R. Powell.

Hydrofluosilicic acid (F.P. 594,590).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Loss of sugar by inversion in [cane] sugar factories in Northern India. C. M. Hutchinson and C. S. Ramayyar (Agric. Res. Inst., Pusa, Bull. 163, 1925, 9 pp.).—Samples of juice from the main channel of the roller mills, juice from the rotating sieves, juice before liming, filtrate from the presses, and juice from the liming tanks, were shown to be considerably infected by bacteria, yeasts, and moulds, which organisms, especially one of the *subtilis* group, readily inverted sucrose. By the use of hypochlorite solution (1 in 25,000) for sprinkling the mills, bagasse carriers, and other parts, a considerable diminution in the invert sugar formation was effected. J. P. Ogilvie.

Modification of Schoorl's method of titrating sugar. I. M. Kolthoff (Chem. Weekblad, 1926, 23, 61—62).—Cuprous salts are quantitatively oxidised to the cupric state by iodine in acid solution in presence of potassium oxalate. The cuprous oxide formed in titration of sugars with Fehling's solution may be determined by addition of sodium carbonate after the ordinary reduction and cooling, adding potassium iodate, hydrochloric acid, potassium iodide, and after 1 min., potassium oxalate, and titrating the excess of iodine with thiosulphate. The sodium carbonate is used to give an atmosphere of carbon dioxide in the flask. S. I. Levy.

See also A., Feb., 121, **Colloidal sugar** (Von Weimarn). 149, **Oxidation of dextrose by means of copper in sodium carbonate solution** (Jensen and Upson); **Behaviour of dextrose and other carbohydrates towards dyes and towards potassium ferricyanide in an alkaline medium** (Knecht and Hibbert). 153, **Polymerisation of β-glucosan. Constitution of synthetic dextrins** (Irvine and Oldham).

Determination of total solids. Röttinger.—See XVIII.

Patents.

Device for treating [extracting] material. P. A. Singer (U.S.P. 1,566,623, 22.12.25. Appl., 12.9.24).—An apparatus for extracting beetroot or the like consists of a vertical cylinder fitted with a reservoir at the bottom. The top of the cylinder is maintained under a partial vacuum, so that the liquor rises about half way up and is there discharged through a pipe fitted with a screen to remove solids. The solid material under treatment is introduced at the top and removed at the bottom. Owing to the reduced pressure the liquid saturates the solid thoroughly and the rate of extraction is greatly increased. T. S. Wheeler.

Adhesive. W. R. Long (U.S.P. 1,564,970. 8.12.25. Appl., 28.2.25).—Maize starch is treated with hydrochloric acid, and the mixture is allowed to remain for 24—48 hrs., after which it is agitated

in a converter wherein it is subjected to heat and steam pressure. J. P. OGILVIE.

Process of manufacturing starch products. P. A. SINGER (U.S.P. 1,564,979, 8.12.25. Appl., 31.1.17).—A liquid spray containing starch is projected into a highly heated reaction chamber in the presence of a reagent, which is allowed to act on the starch particles while these are suspended in a gaseous medium within the chamber J. P. OGILVIE.

Process of making thin boiling starch. J. R. MACMILLAN, Assr. to NIAGARA ALKALI CO. (U.S.P. 1,567,609, 29.12.25. Appl., 11.12.22).—Dry starch is exposed to the action of gaseous chlorine for a short period. G. T. PEARD.

Yeast and alcohol from molasses (G.P. 420,172). —See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Hydrogen ions in brewing processes. G. HAGUES (J. Inst. Brew., 1926, **32**, 8—19).—A new system of wort analysis has been worked out by means of which the composition of a malt wort is shown to differ but little whether made from neutralised carbonate water or from the carbonate water itself, the increased extract in the former case being due to nitrogen compounds and maltose. Although the worts had been previously boiled for two hours, the more acid one showed decided haziness on sterilisation, whereas the more alkaline remained clear. Whether this instability to heat is akin to instability under normal brewing conditions cannot be definitely stated. C. RANKEN.

Analysis of argol and raw materials containing tartaric acid. F. GLASER (Z. anal. Chem., 1925, **67**, 279—281).—Goldenberg's method for the determination of tartaric acid (B., 1908, 245) gives results approximating to the correct figures by a compensation of errors, for part of the tartaric acid fails to precipitate, whilst the precipitate always contains small quantities of acid substances such as acetic acid, phosphates, and pectin-like substances which form salts with the alkali used in the titration. These salts are hydrolysed on heating so that a solution which is neutral to phenolphthalein or litmus in the cold becomes alkaline on warming and low results are obtained. The sensitivity of the litmus paper used also has a marked effect on the titration. A. R. POWELL.

Sugar-inverting bacteria and their industrial application for the preparation of fatty acids, especially lactic, acetic, and butyric acids, and also acetone, ethyl and butyl alcohols, and mannitol. G. MEZZADROLI (Giorn. Chim. Ind. Appl., 1925, **7**, 563—568).—The classification of bacteria which decompose sugar with formation of fatty acids etc., is discussed, and examples are given of the composition of the mixture of products formed by some of these organisms. T. H. POPE.

Fermentation of cacao shell. A. CHURCHMAN (J.S.C.I., 1925, **44**, 540—542 T).—The yield of alcohol from cacao shell by acid hydrolysis, neutralisation, and fermentation was studied. The maximum yield of alcohol was about 3% by weight, the yield being improved by increased pressure, increased time of conversion up to 2 hrs., and increased strength of acid. The best conditions appear to be conversion at 6 atm. pressure with 0·5% sulphuric acid for 1 hr., but the use of cacao shell as a source of "power alcohol" is not a practicable proposition. The experiments indicate the best conditions for obtaining extracts suitable for attack by *Lactobacillus pentoaceticus*, *Bacterium acetoethylicum*, etc. C. O. HARVEY.

Determination of the total solids [of a fermented liquid or carbohydrate solution]. A new micro-method. A. C. ROTTINGER (Oesterr. Chem.-Ztg., 1926, **29**, 1—4).—The extract is determined by absorbing a weighed amount of the solution with filter paper and drying at 100°/20 mm. to constant weight. The filter paper (0·45 mm. thick) is cut into a narrow strip, and then rolled so that it fits into a weighing bottle, 25 mm. diam. and 20 mm. high. Three or four weighing bottles, separated by glass triangles, are placed in a hard glass boiling tube, 30 mm. diam., and 110 mm. high, provided with a rubber bung, through which is passed a calcium chloride tube which terminates in a capillary tube stop-cock. The whole is placed in a boiling water bath for 30 min., then evacuated to 20 mm., and allowed to remain in the bath a further 30 min., after which it is withdrawn and cooled. The bottles are weighed. to 0·1 mg., 5—10 drops of solution are dropped on the roll of filter paper, and the bottles are re-weighed. The above procedure is then repeated till the weight of the bottles becomes constant to 0·2 mg. Duplicate determinations usually agree exactly and the greatest error is 0·2%, whereas the usual methods give errors from +1·5% to —3·3%. The method is unsuitable for solutions containing less than 5% of extract, and solutions containing more than 60% of extract must be diluted. Traces of sucrose (if present) may be inverted during the determination, giving a high extract. This error is less than 0·2%. The temperature must be maintained between 90° and 110° to avoid incomplete drying on one hand, and decomposition on the other. G. T. PEARD.

Volumetric determination of alcohol. G. CHABOT (Bull. Soc. chim. Belg., 1925, **34**, 328—337). —The method of Widmarck (A., 1922, ii, 789) is applied with slight modification to solutions containing 0·3 to 10% of ethyl alcohol, a sample of 0·5 c.c. being taken and the final titration conducted with 0·0175-N thiosulphate solution. The error of the method is ±0·02%, whereas methods depending on distillation and determination of density of the distillate are subject to much larger errors. G. M. BENNETT.

See also A., Feb., 142, **Laboratory pure culture apparatus** (HILDEBRANDT). 147, **Formation of citric acid from gluconic acid by moulds**

(Wehmer); **Formation of citric acid from gluconic acid by *Aspergillus*** (Schreyer). 201, **Do spring waters containing carbon dioxide and hydrogen carbonates activate amylases?** (Loeper and Mougeot); **Spring waters containing hydrogen carbonates and activity of invertase from beer yeast** (Mougeot and Aubertot); **Enzyme from seeds of *Rhamnus*, rhamnodiastase** (Bridel and Charaux). 202, **Maltase from barley malt** (Pringsheim and Leibowitz). 203, **Formation of acetylmethylcarbinol and 2:3-butylene glycol in metabolism of yeast** (Kluyver Donker, and Hooft); **Amino-acid containing sulphur in alcoholic extract of yeast** (Odake). 207, **Synthesis of vitamins by yeasts** (Zajdel and Funk). 212, **Volumetric determination of phosphoric acid in yeast** (Staiger).

High - precision fractional distillation. Peters, jun., and Baker.—See I.

PATENTS.

Drying of agricultural or horticultural products, such as hops, seeds, or the like. V. Elkington (E.P. 245,263, 9.12.24).—Heated dry air is evenly distributed over the top, and heated air, moistened to any desired extent, is distributed at the under surface of the charge of hops in the oast-house. Deposition of water on the upper portion of the change is thereby prevented, thus obviating choking. The drying, which is under delicate control, is a balanced reaction between the top and bottom air, and drying zones can be made to pass up through the charge. A product similar to sun-dried or air-dried hops is obtained. G. T. Peard.

Concentrated enzymic substance. S. A. Waksman, Assr. to J. Wolf and Co. (U.S.P. 1,564,385, 8.12.25. Appl., 11.8.22. Renewed 28.2.25).—A fungus, such as *Aspergillus flavus*, is grown in a suitable culture and extracted with water. A fresh culture is immersed in the extract and the mixture dried under reduced pressure. The product has very high diastatic and proteolytic activity.
T. S. Wheeler.

Manufacture of *n*-butyl alcohol. H. Beinot, Assr. to Commercial Solvents Corp. (U.S.P. 1,565,543, 15.12.25. Appl., 14.4.24).—The addition of calcium or sodium lactate or of a mash undergoing lactic fermentation to a carbohydrate mash undergoing butyl alcohol-acetone fermentation increases the ratio of butyl alcohol to acetone in the resulting product. T. S. Wheeler.

Production of beer. L. Ziselsberger (G.P. 415,903, 20.5.22).—The grits and husk of ground malt are made into a thick mash, which is passed through a mash filter. The flour is mashed separately, and allowed to saccharify at 66—72°, and is then mixed with the thick mash and the whole is allowed to saccharify for a further period. The hops are extracted first with warm water to remove the soft resin, then with boiling water. The mash is boiled first with the boiling-water hop extract until the proteins are coagulated, then the emulsion of the soft hop resin is added and boiling continued for a short time, after which the mash is run into the hop back, whence the sterile wort runs over the refrigerators direct to the fermenting cellar. The maximum extract possible is obtained from the materials by this method. G. T. Peard.

Obtaining yeast and alcohol from a molasses solution. Dresdener Presshefen- u. Kornspiritus-Fabr. sonst J. L. Bramsch (G.P. 420,172, 31.12.21).—A molasses solution is mixed with mineral acid at a temperature below 62°, clarified, filtered, and mixed with the necessary nitrogen-containing compounds. During fermentation, any further increase in acidity, beyond that required for clarification, is prevented by the action of basic nitrogenous substances. Alternatively the molasses solution may be neutralised with mineral acid and the solution then put through a lactic acid fermentation before addition of more mineral acid as described above. Discoloration of the yeast is avoided by the above procedure. G. T. Peard.

Fractionating liquid mixtures (F.P. 595,821).—See I.

Extracting odorous constituents from grain, roots, etc. (F.P. 593,661).—See XX.

XIX.—FOODS.

Bleaching and artifical maturation of flour. Javillier (Ann. Falsif., 1925, 18, 580—595).—The bleaching agents used are enumerated and the usual working quantities of each are given. The bleaching is brought about by the formation of colourless oxy-, chloro-, or nitro-derivatives of carotin, the yellow colouring matter of the flour. The only quantitative difference between treated and untreated flour is that the hydration of the gluten may be slightly higher in the treated flour, and that the iodine absorption number of the ether extract of the original flour (100) is reduced, when nitrous acid is the bleaching agent. Experiments with rats indicate that vitamin-*A* is partially destroyed during bleaching. Vitamin-*B*, however, is not affected. The following amounts of bleaching agent, probably harmless, may be found in the bread, 30 mg. of chlorine, 0·6 mg. of nitrous acid, or 25 mg. of benzoic acid (from benzoyl peroxide) per kg. (100 pts. of flour: 130 pts. of bread). Bleached flour gives, on the average, a 1% higher yield of bread than untreated flour, except when benzoyl peroxide is used, in which case the yield is not improved, but the texture of the bread is. The taste and odour are not altered by the bleaching and probably the most important effect is the artificial maturation which is induced, enabling the miller to reduce the period of storage of the flour. It is recommended that the amounts of bleaching agent permissible should be regulated by law; agents containing chlorine or nitrogen peroxide as active principle should only be used with that fraction of the flour which has been obtained by a high degree

of extraction (milling), say beyond 65%, the miller should state when the flour has been bleached, and indicate the agent used. G. T. PEARD.

Casein content of Danish milk. H. M. HÖYBERG (Z. Fleisch. Milchhyg., 1925, 35, 381—383; Chem. Zentr., 1926, I., 525).—From the results of the examination of milk from three herds (44 cows) and of the mixed milk of a large dairy company, it is concluded that the average casein content of the milk sold in Copenhagen, represents 76% of the total nitrogen of the milk. The figure given by Fleischmann for German milk is 85%.

G. T. PEARD.

Detection of separated [centrifuged] milk in whole milk. E. HEKMA (Jaarversl. Vereen. tot exploit. proefzuivelb. te Hoorn, 1924, 1—3; Chem. Zentr., 1926, I, 528).—The Trypan-blue method described previously (B., 1925, 223) is used, adulteration with separated milk being indicated by the presence of considerable quantities of foam cell membranes in the centrifuge deposit.

G. T. PEARD.

Distinction between buttermilk and soured, separated [centrifuged] milk. E. BROUWER (Jaarversl. Vereen. tot exploit. proefzuivelb. te Hoorn, 1924, 11—13; Chem. Zentr., 1926, I, 528.)—On microscopical examination, cellular structures and butter clumps are found in buttermilk, but are present very rarely or not at all in centrifuged milk, which, however, contains many foam cell membranes produced by centrifuging. G. T. PEARD.

Determination of sucrose in condensed milk and chocolate. G. JORGENSEN (Ann. Falsif., 1925, 18, 517—529).—The Jessen-Hansen method (B., 1923, 1237 A) of determining sucrose is regarded as the most accurate, but in view of the convenience and probable continued use of the polarimetric methods, these are discussed and criticised. Although solution should be effected in warm water, overheating must be guarded against. The advantages gained by control of the p_H value in inversion, particularly in the presence of buffers, is pointed out. For instance, by adding hydrochloric acid to the solution (75 c.c.) until the p_H is 2 (thymol blue as indicator) and then adding a further 1 c.c. of N-acid to bring the value to p_H 1·7, before inversion, the true values of $[\alpha]_D$ may be used in the subsequent calculation of the percentages of sucrose and lactose. In order to obviate the difficulty of accurate calculation of the volume of the precipitate in the case of chocolate, where polarimetric readings at different concentrations have been made, measurements were made by solution, precipitation with lead acetate, filtration, and polarisation, with mixtures of sucrose and cacao, and sucrose and defatted cacao, which established the mean volume of 1 g. of cacao in suspension as 0·93 c.c. in the first case and 0·79 c.c. in the second, so that 0·5 g. of cacao butter will occupy 0·53 c.c. corresponding to d 0·95 at 20°. D. G. HEWER.

Colorimetric determination of the acidity of cheese and whey. B. J. HOLWERDA (Jaarversl. Vereen. tot exploit. proefzuivelb. te Hoorn, 1924, 14—17; Chem. Zentr., 1926, I., 528—529).—The turbidity of whey is removed by shaking with well washed kiesulguhr, which does not alter the p_H, and the colour compensated for by the use of a comparator. The most satisfactory indicators are methyl-red (p_H 4·2—6·3) and neutral-red (p_H 6·5—8·0), the former being more accurate than the latter. The p_H of cheese is determined by placing pieces (about 10 g.) in test-tubes and covering with 30 c.c. of a lactic acid-sodium hydroxide mixture containing varying amounts of lactic acid, and which exerts only a slight buffer action. After 3 min. the p_H of the solution is determined with methyl-red, and compared with that of the original solution. The p_H of the cheese will be equal to that of the solution of which the p_H value does not change during the test. The results are not affected by 1% of sodium chloride. The lactic acid–sodium hydroxide solution is made up by adding to 40 c.c. of 0·1N-sodium hydroxide the following quantities of 0·1N-lactic acid (to give the p_H stated) and diluting to 200 c.c.: p_H 6·0, 40·3 c.c.; 5·6, 40·6; 5·5, 41·0; 5·3, 41·5; 5·1, 42·0; 5·0, 43·0; 4·8, 44·0; 4·7, 45·2; 4·5, 48·0 c.c.

G. T. PEARD.

Fish meals. II. Changes occurring in the water-soluble nitrogen and in the amount of water-soluble phosphorus with different methods of treatment and storage. W. L. DAVIES (J.S.C.I., 1926, 45, 25—29 T; cf. B., 1925, 938).—Samples of five types of fish meals, three of the white variety and two of the yellow, were stored in a cold wet state for 3 weeks, in a warm wet state for 3 weeks, in open vessels for 7 months, in paper bags, and in a warm dry incubator at 38° for 7 months, respectively. Acidity in aqueous extracts of the meals was greatest with meals subjected to the short wet treatments and those in which moulds had grown. Increase of water-soluble nitrogen varied with the type of meal and method of storage. Short wet treatments caused more nitrogen to go into solution, except with a resistant yellow meal and a white "shelly" meal. There was a tendency with long periods of storage for the amount of water-soluble nitrogen to diminish except in the case of a very good white meal, which showed constant values irrespective of treatment. In all cases the amount of nitrogen appearing in the "alcoholic extract" was a higher percentage of the water-soluble nitrogen than in the original meals. The nature of the nitrogen compounds in the aqueous extracts was determined by adding alcohol to a concentration of over 70% and titrating the acid radicals and the free amino-groups, using thymolphthalein and methyl-red as indicators respectively. From the results obtained, conclusions are drawn concerning the process of breaking down which had occurred during keeping. The increase in volatile base content with wet treatment was established, whilst with the other treatments, loss of volatile base to the extent of from 1 to 3·6% of the total nitrogen was shown. The loss was due to escape into the atmosphere and was greatest with the warm dry treatment. Initial acidity had no

effect on the phosphorus entering into solution. Warm dry treatment produced the greatest increase in the amount of soluble phosphorus. Mould growth caused a decrease of soluble phosphorus. Wet treatments caused meals to become unwholesome, but the dry treatments gave no fetid odour even in cases of slight mould growth. Bag storage caused oily meals to become rancid and some white meals to become unwholesome and fetid of odour.

Vitamins in canned foods. IV. Green peas. W. H. Eddy, E. F. Kohman, and V. Carlsson (Ind. Eng. Chem., 1926, 18, 85—89; cf. B., 1924, 195).—The general principle adopted was to feed animals on a diet free from the particular vitamin under investigation until a period of definite decline in weight was recorded, and then to add the peas, and compare the increase in weight for a definite time with that obtained with a standard. Green peas were found to be a valuable source of vitamin-*A*, having in this respect about half the value of butter, the smaller peas being apparently richer in vitamin. The peas were richer in vitamin-*B* than either milk or tomato juice; the more mature peas were richer than the small immature ones, and although there is no appreciable destruction in home cooking or processing of canned peas, the duration and method of blanching is a factor, and the former be reduced as much as possible. With regard to vitamin-*C* peas were found to be one of the best antiscorbutics, ranking with tomatoes and oranges. The antirachitic factor vitamin-D appeared to be absent at the level of intake used. D. G. Hewer.

Vitamin-C in grass. E. Brouwer (Jaarversl. over 1924 Vereen. tot exploit. proef. te Hoorn, 1925, 57—60; Chem. Zentr., 1926, I., 433).—Perennial rye grass is particularly rich in vitamin-*C* and probably contains more than oranges. 1 g. of the fresh grass given daily was sufficient to protect guinea-pigs from scurvy, and had a very favourable influence on the production of bone. The hay or silage prepared from rye grass is poor in vitamin-*C*. C. T. Gimingham.

Jelly strength of pectin jells. G. L. Baker (Ind. Eng. Chem., 1926, 18, 89—93).—The jelly strength was tested by a method in which pressure developed by displacement of air by water in a bottle is transferred to a syringe chamber and manometer so that the syringe plunger is forced into the jelly. The manometer is read when the plunger just breaks through the jelly surface, and the breaking point is recorded as the relative number of cm. of water pressure, and the jelly is then turned out and the operation repeated on the bottom surface. The pressure must always be regulated so that the manometer registers the same figure (*e.g.*, 30) at the end of 1 min. against an unyielding substance. The effect of varying the acidity of the jellies, using $0 \cdot 1N$-solutions of sulphuric, tartaric, and citric acids was to reach an optimum jelly strength, which then decreased with further rise of acidity. The optima were produced with 4·5 c.c. of sulphuric, 9—10 c.c. of tartaric, and 14 c.c. of citric acid. Using 1 g. of pectin a 69·44% sugar concentration produced a maximum jelly strength, which dropped to 66·66 for 2 g. of pectin. A definite pectin-sugar ratio must be maintained for a given p_H value. The optimum pectin concentration is 0·97% of the weight of the finished jelly. Jelly strength is impaired if the pectin is concentrated in the presence of acid before the sugar is added, but a decided increase in invert sugar has no harmful effect. Jelly strength is increased by fall of temperature and by increase in time of standing. D. G. Hewer.

See also A., Feb., 189, **Tryptorhan content of proteins; determination of tryptophan** (Tillmans and Alt). 195, **Influence of certain salts on yield and composition of milk** (Mattick and Wright). 197, **Effect of variation of sodium in a wheat ration** (Olson and St. John); **Rôle of cystine and mineral elements in nutrition** (Woods); **Formation of fat in the pig** (Ellis and Hawkins). 206, **Vitamin-A in beef, pork, and lamb** (Hoagland and Snider); **Chemical nature of vitamin-A** (Drummond, Channon, and Coward). 207, **Technique of testing for presence of vitamin-A** (Drummond, Coward, and Hardy); **Colour reactions associated with vitamin-A** (Fearon); **Antirachitic value of irradiated cholesterol and phytosterol** (Hess, Weinstock, and Sherman); **Technique for studying vitamin-B** (Smith, Cowgill, and Groll). 210, **Phytobiochemical studies** (Zlatarov); **Nitrogen constituents of celery plants on health and disease** (Cooks and Klotz).

Growth of mould fungi on sizing and finishing materials. Growth of mould fungi on steeped wheat flour. Morris.—See V.

Fermentation of cacao shell. Churchman.—See XVIII.

Determination of alkaloids in lupins. Mach.—See XX.

Patents.

Soluble food product, having fresh kola as base. A. and E. Chalas (E.P. 233,695, 30.4.25. Conv., 7.5.24).—Fresh kola nut is freed from oxidising enzymes, *e.g.*, by heating to 80—100° out of contact with air, and an aqueous alcoholic extract of the nut is then prepared. The extract is peptised by hot water and the colloidal solution is mixed with powdered lactose, dried, and ground, granulated, or formed into tablets. C. O. Harvey.

Preserving fruits, vegetables and the like. J. Flach (E.P. 245,671, 2.7.25).—Fruit and vegetables, after treatment with a gas with germicidal and disinfecting properties, such as benzoic acid vapour, carbon monoxide, sulphur dioxide, or formaldehyde, is stored on superposed tray in a chamber through which is passed a current of moist nitrogen or other sterile, non-reacting gas. Two pipes convey the gas into the chamber, the one supplying a mixture of the gas with steam, and the other dry gas; the pipes are fitted with valves operated automatically by

electrical means regulated by a hygrometer within the chamber, so that the moisture content of the gas in the chamber is maintained within limits suitable for the material being stored. L. A. Coles.

Treating vegetable substances. Continuous steam milling of vegetable substances. C. R. Mabee, Assr. to Mabee Patents Corp. (U.S.P. 1,565,282–3, 15.12.25. Appl., 16.8.20).—(a) An apparatus for extracting vegetables with water comprises a digesting tank with an internal screw conveyor for causing continuous movement of the material undergoing extraction. Means are provided for circulating the extracting liquid through the material and for placing the apparatus under reduced or increased pressure; the variations in pressure serve to disintegrate the material and facilitate the extraction. (b) An apparatus for continuously extracting vegetable material with water comprises three cylinders in series, fitted with screw conveyors. In the first the material is thoroughly mixed with water; in the second it is heated and extracted, and in the third the residue is dried. Means are provided for varying the pressure within the first two cylinders. T. S. Wheeler.

Preventing discoloration of canned foods. T. W. Morgan and W. W. Murray, Assrs. to Continental Can Co. (U.S.P. 1,567,163, 29.12.25. Appl., 16.5.25).—Lacquer for applying to the interior of metal containers for canned foods to prevent discoloration of the contents, contains insoluble compounds of such metals as form sulphides which are not dark coloured. L. A. Coles.

Bone meal (Austr. P. 100,717).—See XX.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of nicotine in tobacco. H. Rundshagen (Chem.-Ztg., 1926, **50**, 42).—A modification of Toth's method (B., 1901, 942; 1911, 1084) is described. The difficulties caused by the presence of resins and fats, which form sodium soaps partially soluble in the light petroleum–ether mixture or toluene, thus giving high results, are overcome by the use of calcium hydroxide instead of sodium hydroxide for the liberation of the nicotine. The calcium soaps are very difficultly soluble, and clear solutions are obtained which do not form emulsions during the subsequent titration, which is carried out on a portion of the filtered nicotine solution. The results are about 12% lower than those given by Toth's original method. Similar results are obtained if the tobacco be previously freed from fat with light petroleum, and then it is immaterial whether calcium hydroxide or sodium hydroxide be used for the liberation of the nicotine. E. H. Sharples.

Determination of alkaloids in lupins. Mach (Landw. Vers.-Stat., 1925, **104**, 226—231).—Sparteine is separated from lupinine by distillation in steam and is determined by precipitation with silicotungstic acid. The residue is mixed with gypsum, extracted with chloroform, and after removal of the chloroform, treated with ether. The ether solution is shaken with 5% hydrochloric acid, the acid liquid separated, and the alkaloid finally precipitated with silicotungstic acid. C. T. Gimingham.

Direct crystallisation of citric acid from lemon juice. G. Ajon (Riv. Ital. essenze profumi, 1925, 7, 114—116; Chem. Zentr., 1926, I., 787).—Citric acid crystallises from lemon juice which has been concentrated after removal of iron by precipitation with tannic acid, calcium together with a portion of the magnesium by means of oxalic acid, and potassium by means of tartaric acid. L. A. Coles.

Determination of the alcohol content of tinctures. J. Gadamer and E. Neuhoff (Apoth.-Ztg., 1925, 40, 936—938; Chem. Zentr., 1926, I., 189).—For alcohol determinations, tinctures are diluted with water and distilled. The distillate is treated with potassium carbonate according to the method of Nag and Lal (A., 1918, ii, 411), whereby the hydrate, $4C_2H_5{\cdot}OH,H_2O$, is separated and measured volumetrically. Certain tinctures (*catechu*, *cinnamon*, *Quillaiæ*, *Ratanh.*, *Tormentill.*, *Iodi*) required preliminary treatment before distillation. L. M. Clark.

Albumose-silver. C. Mannich and K. Curtaz (Arch. Pharm., 1925, **263**, 669—674).—Utz (Pharm. Ztg., 1924, 757) states that "protargol" is much superior to other similar protein-silver preparations in respect to fineness of division. The authors cannot substantiate this statement and suggest that the primitive ultra-filters used by Utz have led him into error. Using collodion ultra-filters prepared by Mannich's method (B., 1920, 766 a) six commercial albumose-silver preparations, including "protargol," passed through at approximately the same rate, and similar amounts of silver and protein were found in the filtrates. "Protargol" is, however, superior to other preparations in respect to amount of insoluble matter; it contains traces only, whereas some competing products contain 1·4%. W. A. Silvester.

Thermodynamic consideration of the synthetic methanol [methyl alcohol] process. K. K. Kelley (Ind. Eng. Chem., 1926, **18**, 78).—The free energy of formation and the heat of formation being known for both methyl alcohol and carbon monoxide at 25°, the free energy change involved in the reaction is calculated; from this, the free energy change at any temperature is deduced, allowing for the heat of vaporisation of methyl alcohol since the reaction usually takes place in the gaseous state, and using data for the specific heats given by Partington ("Specific Heats of Gases," 1924). The equilibrium constant $K=P_{\mathrm{MeOH}}/(P_{\mathrm{H_2}})^2(P_{\mathrm{CO}})$ is calculated from the equation $\Delta F=-RT\log_e K$ (ΔF = free energy change), at temperatures from 25° to 600°. The results show that the process is thermodynamically workable at temperatures up to about 450°, with pressures of 100—1000 atm.,

comparing well with the actual working conditions of the process (150 atm. and 300—600°) as given by Patart (cf. Lormand, B., 1925, 525).

B. W. CLARKE.

Influence of chemical constitution on the odour of mustard oils.—G. M. DYSON (Perf. Ess. Oil Rec., 1926, 17, 20—22).—The odours of a large number of pure mustard oils, prepared from thiocarbonyl chloride, have been classified and compared. Strength of odour is generally dependent on volatility; thus lengthening of the carbon chain in normal alkylthiocarbimides causes a decrease from the very penetrating odour of the ethyl compound to the faint odour of the *n*-heptyl derivative. Branching of the carbon chain in secondary and tertiary alkyl compounds leads to a slight increase in the pungency, but the introduction of a phenyl group into the alkyl radical has a very marked effect; β-phenylethylthiocarbimide smells like watercress, of which it is the active odoriferous principle and benzylthiocarbimide has a similar odour, but the α-phenylethyl compound is excessively malodorous. The penetrating smell of phenylthiocarbimide is only slightly modified by the introduction of a single *ortho*- or *meta*-group, whereas substitution in the *para*-position produces an aniseed-like odour which is almost independent of the nature of the substituent group. The effect of the *para*-group is usually predominant when both *ortho*- and *para*-groups are present together, but when two *ortho*- or one *meta*- and one *para*-group are present, the odour is often entirely different from that of either compound produced by *ortho*- or *para*-substitution alone; thus 2:4:6-trimethylphenylthiocarbimide has a delicate flower-like odour. Increase in weight or number of substituent groups usually softens and sweetens the odour and the almost non-volatile nitro- and hydroxy-phenyl derivatives are odourless.

E. H. SHARPLES.

Essential oil of camphor leaves and twigs from Uganda. (Bull. Imp. Inst., 1925, 23, 421—423).—Steam distillation of the air-dried camphor, leaves and twigs yields 2·3% of a pale cream-coloured, almost solid, oil, equivalent to a yield of 2·6% from the moisture-free material. On cooling and pressing, the oil gives about 70% of solid camphor, and including that remaining in the residual oil, the total yield of camphor from the air-dried material may be taken as about 1·8%, which compares favourably with the yields from camphor leaves from other sources.

E. H. SHARPLES.

Essential oil of massoi bark from Papua. (Bull. Imp. Inst., 1925, 23, 423—426).—The bark of *Cinnamomum Massoia*, Schewe. (massoi bark) yields, on steam distillation, 6·3% of a pale yellow, limpid oil, (d_{15}^{15} 1·064, α_D—0·34°, n_D^{20} 1·536) having an odour of cloves and nutmeg and containing 79% of phenols (mainly, or entirely eugenol), 14% of safrole, with 7% of other constituents, principally terpenes.

E. H. SHARPLES.

Medang losoh oil from the Federated Malay States. (Bull. Imp. Inst., 1925, 23, 428—429).—Medang losoh oil distilled from the wood of Cinnamomum parthenoxylon, Meissn., is optically inactive, has d_{15}^{15} 1·103, n_D^{20} 1·538, ester value 0·8, ester value after acetylation 3·5, and congealing point +10°. It is soluble in 3·5 vols. of 90% alcohol at 15° and consists principally of safrole.

E. H. SHARPLES.

Essential oil of *Origanum maru* from Cyprus. (Bull. Imp. Inst., 1925, 23, 426—427).—This pale, brownish-yellow oil, having an odour resembling that of sweet marjoram oil, has d_{15}^{15} 0·904, n_D^{20} 1·4775, α_D^{20} +9° 27′, acid value 4·1, ester value 4·1, and ester value after acetylation 74·5. It is soluble in 1·4 vols. of 80% alcohol at 15° and 2·5% is absorbed by a 5% solution of sodium hydroxide.

E. H. SHARPLES.

Essential oil of *Bifora radians*, M.B. B. PATER (Pharm. Zentr., 1926, 67, 17—18).—The fragrant weed, *Bifora radians*, M.B., which bears a strong resemblance to the coriander plant, contains, when in bloom, a high percentage of a volatile oil with an intense and characteristic odour. The ripe seed is odourless. Owing to the exceedingly volatile character of the oil, distillation of the fresh green plants with adherent blossoms yields only 0·124% of a light green oil having d^{15}_{15} 0·870, α_D −0·2°, n^{20} 1·4624, acid value 23·8, saponif. value 53·3, ester value 29·5; insoluble in 25 pts. of 80% alcohol. On cooling the oil deposits long, white, needles.

E. H. SHARPLES.

See also A., Feb., 114, **Varieties of thorium oxide and their catalytic action in dehydration of alcohol** (LEVI). 134, **Mechanism of catalysis by aluminium oxide** (BOSWELL and DILWORTH); **Catalytic hydrogenation of organic substances** (SCHMIDT). 147, **Ethyl γγ-diethoxyacetoacetate as reagent for synthesis of glyoxalines** (RUGELEY and JOHNSON); **Electrolytic reduction of aldehydes** (SHIMA). 153, **Digitoxin** (WINDAUS and FREESE). 155, **Physiological activity of choline derivatives** (RENSHAW and WARE). 164, **Synthesis of thymol from *iso*propyl alcohol** (BERT and DORIER). 174, **Menthone series** (READ and COOK). 175, **Synthesis of camphor. Catalytic action of reduced copper on borneols** (MASUMOTO). 180, **Synthesis of 5-β-hydroxyethylbarbituric acid and its alkyl derivatives** (CRETCHER, KOCH, and PITTENGER). 182, **Microchemical identification of tyramine and histamine** (VAN ITALLIE and STEENHAUER). 183, **Instability of atropine sulphate** (BODNAR and FERENCZY). 184, **Constitution of corycavidine and corycavamine** (VON BRUCHHAUSEN). 185, **Alkaloids of *Corydalis cava*** (VON BRUCHHAUSEN and SAWAY). 186, **Anthraquinone-β-sulphonic acid as reagent for alkaloids** (ROSENTHALER); **Trypanocidal action and chemical constitution. Arylamides of 4-aminophenylarsinic acid** (KING and MURCH). 187, **Trypanocidal action and chemical constitution. Arsinic acids containing the glyoxaline**

necleus (BALABAN and KING); **Synthesis of 1-phenylbenzthiazolearsinic acids** (BOGERT and CORBITT); **Arsinophenylcinchoninic acid and derivatives** (CALVERY, NOLLER, and ADAMS). 189, **Separation of alcohol from acetaldehyde or acetone** (GORR and WAGNER); **Gasometric determination of acetic anhydride** (WHITFORD). 201, **Enzyme from seeds of *Rhamnus*, rhamnodiastase** (BRIDEL and CHARAUX); **Glucosides in plants hydrolysed by rhamnodiastase** (BRIDEL and CHARAUX). 205, **Pancreatic hormone** (LANGECKER and WIECHOWSKI). 206, **Parathyroid hormone** (COLLIP and CLARK). 208, **Effect of light on alkaloid content of *Lupinus luteus*** (SABALITSCHKA and JUNGERMANN). 211, **Formation and transformation of essential oil in *Pinus strobus*** (PIGULEVSKI and VLADIMIROVA). 212, **Pharacological evaluation of atropine and scapolamine solutions and of their stability** (KÜHL).

High-precision fractional distillation. PETERS, JUN., and BAKER.—See I.

PATENTS.

Pharmaceutical compound [1-methoxymethyl-3 : 7-dimethylxanthine]. FARBENFABR. VORM. F. BAYER U. CO. (E.P. 242,296, 28.10.25. Conv., 29.10.24).—1-*Methoxymethyl*-3 : 7-*dimethylxanthine*, m.p. 168—170°, which resembles caffeine in its physiological action, but is more soluble in water, is prepared by treating theobromine with chloromethyl ether. For example, a mixture of 200 pts. of sodium theobromine, 200 pts. of benzene, and 90 pts. of chloromethyl ether, is heated in an autoclave for 3 hrs. at 120°, the product being extracted with benzene and crystallised from water or alcohol.
L. A. COLES.

Making derivatives of hypothetical imines, including amines and their substitution products. K. F. SCHMIDT (U.S.P. 1,564,631, 8.12.25. Appl., 8.5.25).—When a solution of azoimide in benzene is treated with sulphuric acid, nitrogen is liberated and the benzene is reduced to aniline. If the benzene is replaced by an aldehyde the following reactions occur :—

$$R{\cdot}CHO \rightarrow R{\cdot}CH(OH).N_3 \rightarrow (1)\ RCN \text{ or } (2)\ R{\cdot}NH{\cdot}CHO;$$

which predominates depends on the conditions of the reaction. The formamido-derivative is readily hydrolysed to the corresponding amine, so that the reaction can be applied to replacing an aldehyde group by a nitrilo- or an amino-group. Benzaldehyde, for example, yields benzonitrile or formanilide. Ketones react similarly as in (2) above, acetone yielding methylacetamide and benzophenone giving benzanilide. T. S. WHEELER.

Preparation of alkyl ethers of the morphine series. E. MERCK, Assee. of W. KRAUSS and K. KOULEN (G.P. 418,391, 5.10.23).—The compounds are prepared by treating amino-oxides of the morphine series with alkylating agents, and subsequently splitting off the oxygen atom from the amino-group by the usual method. For example, *N*-sodium hydroxide and methyl sulphate are added slowly to an aqueous suspension of codeine-*N*-oxide, with stirring, and, after solution is complete, excess of sulphur dioxide is passed into the liquor, sodium hydroxide is added, and methylcodeine is recovered by extraction with ether. Methyl- and ethylmorphine are obtained by a similar method from morphine-*N*-oxide, and paracodine from dihydromorphine-*N*-oxide. L. A. COLES.

Preparation of trichloropyruvic acid. S. SKRAUP and F. WOLFSCHLAG (G.P. 418,054, 17.3.22).—Trichloropyruvic acid, which is used in the preparation of pharmaceutical products, is obtained by treating lactic acid or trichlorolactic acid, or concentrated aqueous solutions of these compounds, with chlorine at raised temperatures, *e.g.*, at 110°, in the presence or absence of catalysts, such as iodine or vanadium or iron compounds. L. A. COLES.

Preparation of solid camphene free from chlorine, from pinene hydrochloride. E. MEYER and W. CLAASEN (G.P. 418,057, 20.1.24).—The product is obtained by treating pinene hydrochloride with alkali salts of lower fatty acids, in the presence of phenols or naphthols. For example, a mixture of crude phenol, anhydrous potassium acetate, and pinene hydrochloride is heated, with stirring, under a reflux condenser until the reaction is complete, *i.e.*, for about 8 hrs. Camphene and acetic acid are separated by distillation over an oil bath, and the acetic acid is neutralised with potassium carbonate solution, camphene separating in a 96% yield, as a waxy solid above the solution. L. A. COLES.

Preparation of ketones of the pyridine series. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (G.P. 418,218, 23.11.23).—The products, which serve for the production of compounds of therapeutic value (cf. Benary and Psille, A., 1924, i, 872), are obtained by treating hydroxymethylene derivatives of ketones, or their derivatives, R·CO.CH: CH-OH, where R represents an aryl or alkyl group, with excess of ammonium acetate in the presence of acetic acid, and, after removing any ammonia compound of the hydroxymethylene ketone, separating the pyridyl ketone by the usual method.
L. A. COLES.

Preparation of phenolic derivatives of tetrahydronaphthalene. J. D. RIEDEL A.-G., Assees. of G. SCHROETER (G.P. 417,927, 2.9.22).—Crude or pure tetrahydronaphthalene is chlorinated in the dark in the presence of halogen carriers, the product is sulphonated to a mixture of 1- and 2-chlorotetrahydronaphthalenesulphonic acids, salts of the mixed acids are separated by fractional crystallisation, and converted by treatment at high temperatures with aqueous alkali solutions into the corresponding hydroxysulphonic acids, and these are subsequently hydrolysed to 1- and 2-hydroxytetrahydronaphthalene by boiling with acids. Alternatively, the mixture of 1- and 2-chlorotetrahydronaphthalene can be sulphonated in fractions, sufficient sulphuric acid

being used in the first place to convert the 1-chloro-compound into 1-chlorotetrahydronaphthalene-4-sulphonic acid, the residual 2-chloro-compound being subsequently sulphonated to 2-chlorotetrahydronaphthalene-1-sulphonic acid. For example, a stream of chlorine is passed in the dark into crude tetrahydronaphthalene containing iron wire, at such a rate that a rapid rise in temperature is avoided, until the liquid has gained the theoretical increase in weight. After removal of the catalyst, the product, b.p. 123—125°/11 mm., is separated from unchanged tetrahydronaphthalene and its dichloro-derivative, by fractional distillation. It is then sulphonated with 10% oleum, and the sulphonic acids are converted into their magnesium salts to separate the isomerides, magnesium 1-chlorotetrahydronaphthalene-4-sulphonate and magnesium 2-chlorotetrahydronaphthalene-1-sulphonate being slightly soluble and readily soluble in hot water, respectively. The magnesium salts are converted into the sodium salts, and these are heated with 20% sodium hydroxide at 200° for several hours under pressure, and the products are hydrolysed by boiling with concentrated hydrochloric acid or with moderately dilute sulphuric acid. Polyhydroxy-derivatives are prepared by halogenating the individual hydroxytetrahydronaphthalenesulphonic acids obtained as described above, and continuing the process as before. For example, sodium 2-hydroxytetrahydronaphthalene-1-sulphonate is converted into sodium 2-hydroxy-3-bromotetrahydronaphthalene-1-sulphonate, and, after replacement of the bromine atom by the hydroxyl group, into 2:3-dihydroxytetrahydronaphthalene.

L. A. Coles.

Manufacture of neoarsphenamine [neosalvarsan]. Manufacture of hydrochlorides of 3 : 3′-diamino-4 : 4′-di[hydr]oxyarsenobenzene [salvarsan]. P. A. Kober (U.S.P. 1,564,859–60, 8.12.25. Appl., 12.4.20).—(A) Freshly precipitated neosalvarsan is dissolved almost completely in a strong solution of sodium carbonate, avoiding excess of alkali, and after filtration, the solution is evaporated at 60° at a pressure of 2—10 mm. (B) 3 : 3′-Diamino-4 : 4′-dihydroxyarsenobenzene is dissolved in alkali and the free base is precipitated by neutralisation with hydrochloric acid and then almost completely dissolved in hydrochloric acid solution, excess of acid being avoided. The monohydrochloride is obtained by evaporation under a pressure of 2—10 mm. The dihydrochloride is formed by treating a solution of the monohydrochloride with the theoretical quantity of hydrochloric acid and evaporating as before.

T. S. Wheeler.

Manufacture of alcohols. C. B. Carter and A. E. Coxe, Assrs. to S. Karpen and Bros. (U.S.P. 1,566,818, 22.12.25. Appl., 28.8.22).—Alkyl chlorides are hydrolysed to give satisfactory yields of the corresponding alcohols by heating with aqueous alkali under pressure at 100°, provided the concentration of the alcohol does not rise above 20%. Above this concentration the alcohol and the alkyl chloride react to form the corresponding dialkyl ether.

T. S. Wheeler.

Producing methylals. C. B. Carter, Assr. to S. Karpen and Bros. (U.S.P. 1,566,819, 22.12.25. Appl., 26.7.24).—Methylals are formed in satisfactory yield by heating sodium hydroxide, methylene chloride, and the corresponding alcohol under pressure at 120° for 40 min.

T. S. Wheeler.

Production and purification of hexamethylenetetramine. Recovery of hexamethylenetetramine from mixtures containing ammonium chloride. Production of hexamethylenetetramine and ammonium chloride. C. B. Carter, Assr. to S. Karpen and Bros. (U.S.P. 1,566,820–2, 22.12.25. Appl., 26.7.24).—(A) Hexamethylenetetramine is precipitated almost completely from aqueous solution by saturating the solution with ammonia. (B) Hexamethylenetetramine can be separated from ammonium chloride by saturating an aqueous solution of the mixture with ammonia, when the ammonium chloride is rendered more soluble, and the hexamethylenetetramine is almost completely precipitated. (C) Methylene chloride and ammonia are heated together in aqueous solution, when hexamethylenetetramine and ammonium chloride are formed and are recovered by saturating the solution with ammonia to precipitate most of the hexamethylenetetramine. The ammonia is then distilled off and the residual liquor is cooled, when most of the ammonium chloride separates. The liquid is returned to the process.

T. S. Wheeler.

Extraction of odorous constituents from grain, roots and sweet almonds. R. Gattefossé, and Soc. Franç. de Prod. Aromatiques (anciens Etabl. Gattefossé) (F.P. 593,661, 14.2.25).—Material containing starch and aromatic oils is saccharified and subsequently subjected to alcoholic fermentation, during or after extraction of the oils.

L. A. Coles.

Preparation of colloidal metals or compounds. Königsberger Zellstoff-Fabr. u. Chem. Werke Koholyt A.-G. (G.P. 419,364, 2.3.23).—The metals or compounds are precipitated from solution in presence of products obtained by treating sulphite-cellulose waste liquor with a halogen. Examples given are colloidal copper, soluble copper hydroxide, and a water-soluble bismuth preparation derived from the chloride and containing 45—47% Bi.

C. Irwin.

Regeneration of spent mercury sludge [from the manufacture of acetaldehyde from acetylene]. Verein für chem. Ind. A.-G., Assees. of H. Walter (G.P. 419,435, 22.3.24).—The acid sludge is regenerated by blowing through it the vapour of a suitable liquid. For example, spent sludge from the production of acetaldehyde using a solution of mercuric oxide in 25% sulphuric acid, is regenerated by driving off the aldehyde with a current of steam so that the aldehyde distils over as a 5% solution.

L. A. Coles.

Preparation of stable solutions of therapeutic value, containing calcium and silicon compounds and suitable for injection. Chem. Fabr. Gauff

G.M.B.H., Assees. of M. BERNDT (G.P. 419,676, 5.5.23).—Solutions which can be injected subcutaneously and intramuscularly for treatment of tuberculosis and which are not decomposed by the carbon dioxide of the body, thus avoiding tissue necrosis, are prepared by treating a feebly acid colloidal solution of silicic acid with a feebly acid solution of a calcium salt. For example, a solution of pure sodium silicate, in water distilled from silica vessels, is treated with pure hydrochloric acid until it is faintly acid, and to the mixture is added a similarly acidified solution of calcium glycerophosphate. The formation of insoluble calcium silicate is avoided. If necessary, an aqueous solution of sodium monomethylarsinate may be added. E. H. SHARPLES.

Production of stable cod liver oil emulsions which deposit no sediment on keeping. J. SCHWARZKOPF (G.P. 419,731, 1.3.24).—Sedimentation of cod liver oil emulsions is prevented by the addition of hydrogenated fat, *e.g.*, 1—2% of hydrogenated marine animal oil is added to an emulsion containing 40 pts. of cod liver oil, 0·5 pt. of gum tragacanth, 0·5 pt. of gum arabic, and 59 pts. of water. L. A. COLES.

Preparation of 1-phenyl-2:3-dimethyl-4-dimethylamino-5-pyrazolone. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (Swiss P. 108,599, 29.9.23. Conv., 2.3.23).—1-Phenyl-2:3-dimethyl-4-dimethylamino-5-pyrazolone is obtained by reducing the nitroso-group in 1-phenyl-2:3-dimethyl-4-nitroso-5-pyrazolone to the amino-group by treatment with hydrogen sulphide, and adding the solution, without separation of the amino-compound, to a hot mixture of formaldehyde and formic acid containing, *e.g.*, 32% of formaldehyde and 81% of formic acid. After heating at 100° for 5 hrs. under a reflux condenser, the solution is made alkaline with sodium hydroxide, and the product is extracted with benzene. The small quantity of the 4-formamido-compound present in the crude product remaining after removal of the solvent, is converted into the 4-dimethylamino-compound, with separation of resin, by heating with dilute sulphuric acid and 30% formaldehyde. L. A. COLES.

Preparation of mercury derivatives of organic compounds. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (Austr. P. 99,678, 11.5.23. Conv., 12.5.22).—Mercurated compounds of therapeutic value are produced by forming additive products of mercuric oxide or mercury salts with aliphatic or aromatic compounds substituted with unsaturated alkyl groups, followed if necessary by the introduction into the molecule of residues capable of rendering the products soluble in water. For example, a mixture of methyl alcoholic solutions of mercury acetate and eugenoleacetic acid deposits on keeping crystals of the *compound*,

$$CO_2H\cdot CH_2\cdot O\cdot C_6H_3(OMe)\cdot CH_2\cdot CH(CH_2OMe).(HgO\cdot COMe),$$

m.p. 172°. An aqueous solution of sodium *p*-allyloxybenzenesulphonate, obtained from allyl bromide and sodium *p*-phenolsulphonate, on shaking with mercuric oxide, yields *sodium mercuri*-p-*allyloxybenzenesulphonate*. Other mercurated compounds, obtained by the action of mercury acetate in aqueous or methyl alcoholic solution, include the *mercury compound*, m.p. 185°, from 3-*bromo* 4-*allyloxybenzene*-1-*carboxylic acid* (m.p. 179°, prepared by the action of allyl bromide upon a solution of methyl-3-bromo-4-hydroxybenzene-1-carboxylate in the presence of sodium and methyl alcohol, and hydrolysis of the product); *mercuri*-3-*chloro*-4-*allyloxybenzene*-1-*carboxylic acid*, m.p. 157°; *mercuri*-3:5-*dibromo*-4-*allyloxybenzene*-1-*carboxylic acid*, m.p. 175°; the *mercury compound*, an oil setting to a crystalline mass, from *salicylallylamide-acetic acid* (m.p. 120°, obtained by the action of chloroacetic acid upon salicylallylamide in the presence of boiling sodium hydroxide solution); the *mercury compound*, an oil, from the *p*-phenolsulphonate of 4-*diethylaminoethoxy*-5-*methoxy*-1-*allylbenzene* (b.p. 158°/10 mm., obtained by the condensation of eugenole with chloroethyl-diethylamine in the presence of sodium and alcohol); the *mercury compound* from the sulphate of N-*methyl*-8-*allyloxytetrahydroquinoline* (b.p. 194—196°/12 mm., obtained by the action of allyl bromide upon a solution of *N*-methyl-8-hydroxyquinoline in dilute sodium hydroxide); the *mercury compound* from the tartrate of *allyl p-diethylaminoethoxybenzoate* (an oil of b.p. 210°/10 mm., obtained by treating allyl *p*-hydroxybenzoate, m.p. 105°, with chloroethyl-diethylamine in the presence of sodium and alcohol); the *mercury compound*, an oil, from the tartrate of *diethylaminoethyl* p-*allyloxybenzoate* (b.p. 220°/15 mm., obtained by the condensation of chloroethyldiethylamine with *p*-allyloxybenzoic acid in the presence of sodium ethoxide, *p*-allyloxybenzoic acid, m.p. 164°, being obtained by the condensation of methyl *p*-hydroxybenzoate with allyl bromide and hydrolysis of the product. L. A. COLES.

Preparation of bone-meal for medicaments. F. RASKA (Austr. P. 100,717, 17.7.22. Conv., 26.5.22).—Substances containing vitamins are mixed with prepared bone-meal, *e.g.*, fresh, aseptic bones are carefully degreased with light benzine or benzine vapour, then ground and treated with dilute sulphuric acid (*d* 1·26—1·32), so that only little free phosphoric acid is formed. The product, the gelatin content of which is not affected by the above treatment, after keeping for 2—3 days at ordinary temperature, is mixed with malt, malt germs, or the germs of cereals and used as an addition to feeding stuffs for animals suffering from rickets or bone softening. E. H. SHARPLES.

Process of preparing propylenethiohydrin or propylene-ethylenethiohydrin. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING, Assees. of G. KRÄNZLEIN and M. CORELL (U.S.P. 1,570,262, 19.1.26. Appl., 7.8.22).—See E.P. 185,403; B., 1923, 118 A.

Synthetic manufacture of methanol [methyl alcohol]. BADISCHE ANILIN- U. SODA-FABR., Assees. of A. MITTASCH and M. PIER (U.S.P. 1,569,775, 12.1.26. Appl., 4.9.24).—See E.P. 240,955; B., 1925, 1012.

Manufacture of derivatives of 4-hydroxypiperidines. Soc. of Chem. Ind. in Basle, Assees. of H. Staudinger (U.S.P. 1,567,200, 29.12.25. Appl., 28.3.25).—See E.P. 232,207; B., 1925, 942.

Manufacture of formamide. Badische Anilin- u. Soda-Fabr., Assees. of R. Wietzel (U.S.P. 1,567,312, 29.12.25. Appl., 20.5.25).—See E.P. 240,087; B., 1925, 971.

Injection [colloidal magnesium] for hæmorrhoids. J. Kato (U.S.P. 1,567,847, 29.12.25 Appl., 4.10.20).—See E.P. 202,743; B., 1923, 1044 A.

Colloids (G.P. 416,062).—See I.

Dinaphthomethylamines (Swiss P. 108,704 and 109,066).—See IV.

Converting vegetable fibre to pulverised form (U.S.P. 1,565,864).—See V.

Abietic acid (E.P. 244,980).—See XIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Sensitisation by nuclei of silver sulphide. S. E. Sheppard (Brit. J. Phot., 1926, 73, 33—34).—The author's theory of the sensitising action of silver sulphide centres differs from that of P. E. Henry (Rev. Franç. Phot., 1925, 6, 292) in that the nuclei are supposed only to affect the orientation of the photochemical decomposition, and not to increase the photochemical sensitivity of the silver halide. It is a matter of sensitisation for development rather than for exposure, the centres operating by concentrating the photochemically reduced silver atoms about themselves so as to form a nucleus large enough to induce developability. This is supposed to result from a sphere of disorientation of the lattice around the included silver sulphide nuclei, giving rise to a gradient of flow of absorbed radiation centripetal to the nuclei, so that the final photochemical decomposition is effected in their neighbourhood, where the ionic deformation is greatest. It is possible that this centripetal flow of radiation towards the nuclei is intensified, in the region of absorption of light by silver bromide, by a centrifugal irradiation outwards from the highly absorbing silver sulphide nuclei. Evidence for an optical sensitisation, such as would be expected in the case of ionic deformation, has been obtained by E. J. Bullock, who found that bathing a plate in very dilute sodium thiosulphate, with subsequent formation of silver sulphide, leads to deep red and infra-red sensitising, for long exposures. With silver bromide and silver sulphide precipitated together, *X*-ray analysis gives definite evidence of lattice interpenetration, with actually a very slight shift of the lattice interval of the silver bromide. W. Clark.

Action of dyestuffs on the sensitivity of silver bromide–gelatin. L. Gorini and A. Dansi (Riv. Fotigraf. Ital., 1925, 8 pp.; Chem. Zentr., 1926, I., 559).—Lüppo-Cramer's hypothesis, that the desensitising action of dyestuffs is due to the presence of a number of amino-groups in the dye molecule, was tested by bathing silver bromide–gelatin plates in 1 : 20,000 solutions of the following dyes: Safranol, Safraninone, Phenosafranine, Tolusafranine, the amino- and diamino-derivatives of Safranine, Naphthosafranine, Pinakryptol, Pinakryptol Green, Pinakryptol Yellow, and Aurantia. After drying, the plates were exposed to a spark spectrum for varying times, and the blackening curves for individual colours of the spectrum were plotted. Plates were arranged in series of increasing desensitisation and the results obtained were not in accord with Lüppo-Cramer's theory. It is concluded that it is not possible to give an explanation of desensitisation based only on the chemical constitution of the desensitiser. W. Clark.

Photochemical studies. VI. Mechanical actions on the photographic plate. A. Reychler (Bull. Soc. chim. Belg., 1925, 34, 294—296).—If lines are traced with a blunt glass point on the emulsion surface of a plate which is subsequently developed and fixed, a black image is formed. Microscopical examination of the wet plate shows a channel, edged by elevations, where the point has pressed. The black image is at the bottom of the hollow part only. If the plate is exposed uniformly after inscribing and before developing, the image is less dense than the background; the bottoms of the channels are darkened, but the raised edges are free from developed silver. The pressure has desensitised these edges to subsequent exposure, and it is assumed that this is due to a lack of optical homogeneity introduced at the displaced edges of the line. If the plate is exposed uniformly to light before rubbing, the image is darker than the general ground, the bottom of the hollows being darker than, and the edges of the same density as, the rest of the plate. This is merely due to the summation of two effects which give density. The author's experience agrees with that of P. Wulff (cf. B., 1925, 477) that it is pressure complicated by friction which makes the emulsion developable. W. Clark.

Colloid chemistry and printing. Treichel.—See VI.

Patents.

[Photographic] reversal process. J. G. Capstaff, Assr. to Eastman Kodak Co. (U.S.P. 1,564,753, 8.12.25. Appl., 14.11.23).—A silver image is formed in the usual manner on a photographic plate containing sensitised silver bromide, and, after washing, immersed in a bath of potassium ferricyanide, potassium bromide, and water. The metallic silver on the plate is converted into silver bromide in a form relatively insensitive to light. The plate is then exposed to light for a short time, when the sensitive silver bromide present, which forms a positive of the initial image, is attacked and on developing and fixing yields a silver positive image. The process can also be applied to the production of coloured images. T. S. Wheeler.

Preservative for motion-picture films. J. I. CRABTREE, Assr. to EASTMAN KODAK Co. (U.S.P. 1,568,658, 5.1.26. Appl., 3.3.25).—The flexibility of the films is preserved by immersing them in a bath containing glycerin and ethylene glycol.

L. A. COLES.

Rendering gelatin insoluble and producing images in relief. S. DE PROCOUDINE-GORSKY and N. POZNIAKOW (F.P. 589,422, 19.11.24).—The purest gelatin is mixed with a silver halide which contains no colloid. After reduction of the silver salt by exposure, the gelatin in the places where reduction occurred is insoluble in water, while where the silver salt was not reduced the gelatin is soluble in water. To produce images in relief a gelatin silver bromide paper is exposed, developed with pyrogallol or pyrocatechol, and the image brought on to a polished surface of glass, metal, or celluloid, washed with water at 45—55° and fixed after the paper has been stripped off. S. BINNING.

Light-sensitive preparations for photographic printing. BADISCHE ANILIN UND SODA FABR., Assees. of W. FRANKENBURGER (G.P. 416,995. 10.8.24).—Iron carbonyl is used as the light-sensitive substance, and the image is fixed by treatment with liquids such as water, benzol, alcohol, or benzine, which remove the unexposed iron carbonyl. The sensitive substance is coated, with or without a binding medium, on paper, glass, etc.

W. CLARK.

Reduction of photographic negatives, diapositives, films, bromide and gaslight images. P. PLAGWITZ (G.P. 417,872, 16.8.24).—The image is treated with a mildly-acting reducer and simultaneously or previously with alkali chromate or dichromate. For example, the image is treated with a solution of iodine, potassium iodide, and dichromate together, or else first with dichromate followed by iodine-potassium iodide. Sugar may be present in the reducing solution. W. CLARK.

Recovery of silver from precipitates in photographic solutions. J. G. SEGARRA (F.P. 591,546, 12.1.25).—The precipitate is treated with alkali polysulphides or other substances which readily yield sulphur. Silver sulphide is formed, and is converted into the metal by fusion with sodium carbonate or sodium nitrate, or into a silver-lead alloy by fusion with lead oxide. W. CLARK.

Fluorescent screen for use with *X*-rays. R. JAHODA and L. LILIENFELD (Austr. P. 99,844, 29.7.22).—Zinc sulphide precipitated from solutions obtained by treating zinc with insufficient acid to dissolve it completely is mixed with small quantities of salts of metals such as silver, bismuth, and tungsten, or mixtures of these salts, and heated to redness in the absence of air. The product is separated into fractions which show strong and feeble fluorescence respectively under *X*-rays, and the strongly fluorescent fraction is freed from coarser particles by elutriation and cemented on to a screen by means of suitable binding material. The screens are only feebly phosphorescent, and give strong images. L. A. COLES.

Colour photography. L. D. MANNES and L. GODOWSKY, JUN. (E.P. 245,198, 1.10.24).—See U.S.P. 1,516,824; B., 1925, 115.

Photographic pigmentary printing paper. J. SURY (U.S.P. 1,571,103, 26.1.26. Appl., 26.5.24). —See G.P. 405,494; B., 1925, 267.

XXII.—EXPLOSIVES; MATCHES.

Safety in the manufacture of nitric, sulphuric, and mixed acids, and ammonium nitrate, as used in explosives. S. H. KERSHAW (Ind. Eng. Chem., 1926, **18**, 4—9).—Several devices are described to protect workmen in acid plants from being splashed by acids. Among them may be mentioned a splash-pot for use on the discharge end of pipes through which acid is blown into open tanks, a lead spray shield to be attached to the stems of acid valves, a bridle to prevent stoneware cocks from being forced out by the head of acid, and a trap for use in drawing off nitroglycerin waste acid from open tanks. The chief risks in the manufacture of ammonium nitrate are of fire, of decomposition of an acid charge, and of explosions caused by the presence of carbonaceous matter. Most fires occur during storage and many can be traced to the artificial heating required owing to the hygroscopicity of the material. In stores heated by steam pipes, the latter should pass along the walls and not under the floor and should be fenced off to prevent the nitrate bags or casks from coming into contact with them. Decomposition during evaporation is due to the charge being acid instead of neutral and is not serious if organic matter is absent. An acid charge may be detected by the liquor acquiring a lemon-yellow colour. When this colour is noticed powdered chalk should be sprinkled on the liquor till neutrality is attained, as otherwise a rapid decomposition may set in with generation of enough heat to ignite surrounding wooden structures. Explosions due to carbonaceous matter occur most frequently during evaporating. Most of the organic matter forms a scum on the surface of the liquid and is removed, but if any remains and reaches the steam coils, there is considerable risk of explosion when the liquid is drawn down. The steam should be shut off before drawing down takes place and a bleeder valve opened to ensure that the coils are not heated by steam leaking past the valves.

S. BINNING.

Influence of sunlight on trinitrotoluene. D. LODATI (Giorn. Chim. Ind. Appl., 1925, 7, 572).—The author refutes Krauz and Turek's statement (B., 1925, 479; cf. Wichert and Donat, B., 1925, 571) that, after exposure to sunlight, trinitrotoluene exhibits increased sensitiveness to shock owing to the auto-formation of picric and trinitrobenzoic acids. In diffuse light trinitrotoluene undergoes alteration in its molecular composition, nitrous vapours being generated. T. H. POPE.

Production of coloured smoke signals. A. B. RAY (Ind. Eng. Chem., 1926, 18, 10—17).—A thick black smoke was obtained by the free combustion of a mixture containing hexachloroethane 60·5, magnesium powder 18·6, and naphthalene 20·9%. The effectiveness was increased by replacing the naphthalene by anthracene (8 pts. of anthracene for 9 pts. of naphthalene). The size of the magnesium particles has an influence on the rate of burning, which is slower with coarse particles. The mixture is ignited by a mixture of 7 pts. of reduced iron and 8 of potassium permanganate. Coloured smokes are produced by volatilising organic dyes by burning a mixture of potassium chlorate, lactose, and a dye in a canister pierced with a few holes. Ignition is effected at each by a quick match and igniting mixture. For red smoke the following percentage mixture is recommended. Paranitraniline Red 65, lactose 20, and potassium chlorate 15; for yellow, Chrysoidine Orange 9%, Auramine Yellow O 34, potassium chlorate 33, lactose 24; for blue, indigo (synthetic) 40, potassium chlorate 35, lactose 25%. No satisfactory green smoke was found.

S. BINNING.

PATENTS.

Manufacturing adhesive blends of sodium nitrate and ammonium nitrate for explosive purposes. W. M. DEHN (U.S.P. 1,568,324, 5.1.26. Appl., 13.10.24).—Mixtures of sodium nitrate and ammonium nitrate are changed to sludges by heat and then cooled rapidly to uniform conglomerates which are subsequently pulverised.

S. BINNING.

Explosive mixture. A. WOHL (G.P. 419,127, 8.8.24).—An explosive, which has the high brisance of carbon explosives, is prepared at the place of application by adding to solid transformation products of acetylene a solution of ammonium nitrate in strong nitric acid with or without addition of other substances. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Analysis of soapy solutions of phenols [disinfectants]. F. DI STEFANO (Annali Chim. Appl., 1925, 15, 573—580).—The various methods proposed for analysing disinfectants composed of solutions of soap and phenols, particularly cresol, are discussed. The analysis of a sample supposed to be prepared from castor oil was effected as follows: 20 g. of the liquid were dissolved in 60 c.c. of water, acidified with sulphuric acid, and distilled in a current of steam to eliminate the cresol. The residual fatty acids were cooled to cause them to separate better and, after removal of the supernatant water, were dissolved in sodium hydroxide solution. The filtered solution was treated with excess of dilute sulphuric acid and the deposited fatty acids were filtered off, freed from sulphuric acid by repeated washing with hot water, and dissolved in ether. The residue left on distillation of the ethereal solution was heated in a steam oven for about an hour and then filtered through a dry filter. The acetyl value of the fatty acids thus prepared was found to be 49, which indicates that the antiseptic was made from oleine, since a laboratory preparation, made from castor oil, sodium hydroxide, and cresol, gave, when tested as above, the value 145·5. The faulty solubility of the sample examined is thus due to the use of oleine.

T. H. POPE.

Measuring the smoke pollution of city air. J. S. OWENS (Analyst, 1926, 51, 2—18).—Smoke pollution is measured either as deposit from the air or as impurity suspended in the air. Suspended matter may be measured by impact methods, where efficiency is a function of the velocity of the jet, and consequently any adhesive applied to the impact surface to retain the dust is liable to be blown away from the point of impact; by filtration methods, which require long periods of measurement; by agitation with water, as in Palmer's apparatus, where insoluble dust is washed out; by condensation, where the number of drops of moisture settling on dust particles from a known volume are counted, so that insoluble non-hygroscopic dust is ignored; by electrical precipitation, which is satisfactory but requires a source of electricity and usually a high voltage; or by the ultramicroscope, where counting is difficult and a very small volume of air is used. In settlement methods a fixed volume of air must be enclosed, and all the dust allowed to settle, as in the settlement dust counter described in the Report of the Advisory Committee on Atmospheric Pollution, for the year ending March 31, 1924. The methods adopted by the Advisory Committee are of three types: the deposit gauge, the automatic filter or recorder, and the dust counter. Fifty-eight standard deposit gauges are in operation, and the collected deposits are systematically analysed. In the automatic filter 2 litres of air at a time are filtered through thin paper discs of $\frac{1}{8}$ in. diam., arranged on a turntable, and the shades obtained are matched against standards. In the jet dust counter devised by the author, the jet of air, excessively thin and flat (1 cm. wide and 0·1 mm. thick), is caused to strike a microscope cover-glass at such a high velocity that condensation of water on the dust particles causes wetting of the glass, and consequent adhesion of the dust. A damping chamber may be used if necessary. The linear trace of dust is mounted, record downwards, over a paper ring on a microscope slide. Soluble salts present in the air may be crystallised by using a large volume of air (say 1000 c.c.) and slightly warming the damping chamber, so that the condensed water is blown out sideways, and as the heads of the streams dry immediately, the crystals are left and the salts can be identified microscopically. Oil drops may also be separated from the record by suitable manipulation, and acid and alkali changes can be followed. D. G. HEWER.

Separation of small quantities of calcium from large amounts of magnesium in water. H. NOLL (Chem.-Ztg., 1925, 49, 1071—1072).—Precipitation of calcium as oxalate from dilute acetic

acid solutions yields satisfactory figures only when less than 0·125 g. of magnesium is present in 200 c.c. of the solution (cf. Rodt and Kindscher, B., 1925, 155; Heilingötter, B., 1925, 430; Luff, B., 1925, 429). Too large an excess of ammonium oxalate must be avoided and re-precipitation of the calcium oxalate is essential. It is immaterial whether the precipitate is filtered off immediately or after 24 hrs.

A. R. POWELL.

Amounts of soap and "builder" necessary to soften water of different degrees of hardness. H. B. ROBBINS, H. J. MACMILLAN, and L. W. BOSART (Ind. Eng. Chem., 1926, 18, 27—29).—Experiments with waters of known hardness, both on a laboratory and a laundry scale, showed that if soap and soda are added together, the cost of completely softening the water is practically independent of the relative proportions used. For maximum economy the soda should be added to the water and the mixture agitated for 1 min. before introducing the soap. The amounts of soda ash and soap necessary for the most economical treatment of waters of varying hardness are tabulated. D. G. HEWER.

Oxidisability of water as measured by the Kubel-Tiemann method and the determination of the "chlorine number." K. KEISER (Gas- u. Wasserf., 1926, 69, 41—43, 65—69).—The determination of the oxidisability of water containing organic substances can generally be carried out by Kubel's permanganate method, or by the chlorination method of Froboese (Arb. Reichsges.-Amt, 1920, 52, 211). If, however, humic substances or protein degradation products are present, the chlorine method is the only accurate one, permanganate having no effect on these. Comparative figures are given for water containing urea and for river waters contaminated by sewage. Treatment of the water with small quantities of chlorine, sufficient to destroy bacteria, does not affect the oxidisability as measured by the permanganate consumption. The efficiency of the filtration process is increased and the life of the filter-beds prolonged by a preliminary chlorination of the water. B. W. CLARKE.

Determination of permanent hardness of water by Pfeifer and Wartha's method. A. LUMSDEN-BEDINGFELD (J.S.C.I., 1926, 45, 36 T).—According to Pfeifer and Wartha's method (Z. anal. Chem., 1902, 41, 198) waters containing magnesium salts in addition to calcium, are concentrated after addition of standard solutions of sodium hydroxide and sodium carbonate, then filtered, and the filtrates titrated. During the filtration the filter paper absorbs sodium hydroxide from the solution, and the permanent hardness figure is thereby considerably increased above its true value.

New class of disinfectants: chlorinated sulphonic acids of high molecular weight [from sulphite-cellulose waste lye]. S. HILPERT (Biochem. Z., 1925, 166, 89—94).—By chlorination of sulphite-cellulose waste lye, which consists mainly of ligninsulphonic acids, a product, containing 25—28% Cl is obtained, which is odourless, and possesses strong disinfectant properties. It is soluble in water, but only slightly soluble in alcohol and ether. Since the bactericidal action is due to liberation of hydrochloric acid by hydrolysis at the surface of the bacteria, the disinfectant is inactive in serum. Products similar in action have been obtained by chlorination of naphthalenesulphonic and anthracenesulphonic acids.

E. C. SMITH.

Antiseptic power of chloropicrin. H. VIOLLE (Compt. rend., 1926, 182, 290—292).—The effect of chloropicrin on pathogenic bacilli has been investigated, by adding a few drops of a saturated aqueous solution to broth, before or after the latter had been inoculated with microbic cultures. The preventive power, or quantity which must be added to a sterile solution in order to maintain its sterility after impregnating with microbes, is of the order 1/41,000 to 1/82,000. The growth of microbes is prevented by 12 mg. of chloropicrin per litre of broth, except for sporulated microbes which require double the quantity. The destructive power is developed in about 1 hr. for ordinary and 4 hrs. for sporulated bacteria. Evaporation of the chloropicrin must be prevented. W. HUME-ROTHERY.

Photometric methods for studying and estimating suspensions of dusts, fumes, and smokes. P. DRINKER, R. M. THOMSON, and J. L. FINN (J. Ind. Hyg., 1925, 7, 567—576).—Photometric methods provide the most rapid means of estimating the concentration of suspended matter in the air, although the readings obtained cannot always be converted into concentrations of the particular suspension. Reliable information is, however, obtained of the size and degree of dispersion of the particles, and the methods are equally applicable to true colloidal suspensions and to unstable suspensions, such as silica dust in air. The Tyndallmeter described by Tolman and Vliet (B., 1919, 275 A., 306 A, 391 A) with some slight modifications is the most suitable instrument for field work. The iron pipe should be replaced by hard rubber painted dead black inside, and Edison alkaline batteries, which are very light, are the best means of supplying current for the lamp and for a small suction fan for drawing the sample of air or gas through the examination chamber. The theory of photometric measurements of the concentration of suspensions is briefly discussed. A. R. POWELL.

Relative toxicity of arsenates of calcium. HENDRICKS, BACOT, and YOUNG.—See XVI.

PATENTS.

Determining the bacterial content of the air. LURGI APPARATEBAU-GES. m.b.H. (G.P. 420,725, 20.6.24).—A definite volume of air is passed through a precipitating field between at least two electrodes of opposite polarity, the precipitating surfaces, *e.g.*, the collecting electrodes, being coated with a nutrient fluid or gelatin upon which the precipitated germs or bacteria develop. The rate of growth of the bacteria enables their absolute amount in a definite volume of air to be determined. J. S. G. THOMAS.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 2, 1926.

I.—GENERAL; PLANT; MACHINERY.

Distillation and rectification. L. Gay (Chim. et Ind., 1926, **15**, 3—13, 173—183).—The study of the problem of the complete separation of a mixture of three constituents (*ibid.*, 1923, 811, 1026) is extended to the case of four or more constituents. As in each step in rectification the pure constituents may be separated as either vapour or liquid, the number of ways of proceeding increases rapidly with the complexity of the mixture. A quaternary mixture may be represented in space by a regular tetrahedron on which the composition of the liquid or vapour at any point in the first rectification column may be represented. The calculation of the minimum heat required to operate a column rectifying a quaternary mixture is very complex but may be simplified in the case of a mixture of four successive members of a homologous series, for which the molecular latent heats of evaporation at a given temperature are approximately the same. For ternary mixtures of this type a graphical method is developed for calculating the relation between heat flow and the composition of either phase at any point in the two columns by means of the representation of composition as a triangle.

C. Irwin.

Determination of the size and number of pores of porous filtering plates. H. Ruoss (Chem.-Ztg., 1926, **50**, 83—84).—The size of the pores of a porous plate may be determined roughly by fixing the plate to a glass tube by means of a rubber band, saturating it with water, suspending it just below the surface of the water in a glass vessel, and observing the pressure required to be applied to the upper side of the plate to cause bubbles of air to form on the lower. Unless the pores are of approximately equal size and regular in cross-section this method gives simply the diameter, at the surface, of the largest pores. The average diameter of the pores is ascertained by measuring the rate of flow of water through the plate under constant pressure, the weight of water flowing through the plate being directly proportional to the pressure.

A. R. Powell.

Patents.

Separation of vaporised organic substances from gaseous mixtures. J. J. V. Armstrong. From N. V. Algem. Chem. Productenhandel (E.P. 245,856, 21.9.25).—An adsorbent such as carbon is contained in a gas-tight casing and is subdivided into thin layers by a number of pipe coils which occupy 8—12% of the internal space. A cooling medium is passed through the pipe coils during adsorption and a heating medium in the same coils is used in conjunction with a vacuum over the adsorbent for regeneration.

B. M. Venables.

Treatment of liquids, gases, or solutions with active carbon. H. E. Potts. From N. V. Algem. Norit Maatschappij (E.P. 245,912, 22.2. and 9.5.24).—Liquids or gases are treated with carbon which is much more active than Norit, and having a true sp. gr. (determined in ether) of not less than 2·0, a heat of adsorption of benzene of more than 40 cal. per g., and one or more of the following properties :—elementary carbon not less than 97% calculated free of ash and moisture, an organised structure derived from vegetable raw material, an apparent sp. gr. of less than 0·1, a moisture adsorption of not less than 20% from an atmosphere at ordinary temperature and relative humidity of 60% ; 0·5 g. of the carbon adsorbs more than 80% of the iodine in 50 c.c. of 0·1*N*-solution. The active carbon is prepared from pine wood or other vegetable matter by known means (cf. E.P. 228,582, 228,812, 228,954, and 230,293 ; B., 1925, 275, 346) in a current of gases (*e.g.*, superheated steam) which is adjusted to such a rate of flow as to draw off the activated carbon of very low apparent sp. gr., leaving the unactivated carbon behind in the retort.

B. M. Venables.

Machines for emulsifying and disintegrating solids suspended in liquids. G. C. Hurrell (E.P. 245,929, 2.1.25).—In a high-speed emulsifying machine, to prevent the induction of air, the material is introduced into the shearing zone through a hollow shaft and radial passages in the rotor, and the rate of flow of material is regulated by restriction of the outer ends of the radial passages, one means of adjustment being grub screws which partially obstruct the passages. The machine may conveniently have a vertical shaft, the hollow end of which extends downwards into a supply reservoir.

B. M. Venables.

Utilisation of products of combustion in steam generators. G. and J. Weir, Ltd., and J. G. Weir (E.P. 245,960, 25.2.25).—Products of combustion containing a large amount of water vapour are passed through an ordinary boiler and then through a low-pressure boiler, and the products of combustion under both boilers are maintained at considerable pressure (say 10 lb. per sq. in.) so that the water vapour in them will be condensed while under the low-pressure boiler and give up its latent

heat. The pressure in the flues is maintained by a fan at the inlet and a turbine at the outlet, and the latter may drive the former. B. M. VENABLES.

[Electrostatically] separating suspended particles from flowing gases or vapours. LODGE-COTTRELL, LTD. From LURGI APPARATEBAU-G.M.B.H. (E.P. 246,046, 5.8.25).—An electrostatic gas cleaner is arranged with horizontal electrodes, and the collecting electrodes are composed of a liquid, preferably flowing at about the same rate as the gases. B. M. VENABLES.

Process for making a reflocculated product. G. W. ACHESON (U.S.P. 1,563,713, 1.12.25. Appl., 22.11.24).—Fuller's earth, china clay, or the like is deflocculated by grinding with a deflocculating agent and a limited quantity of water, *e.g.*, 28%, as described in U.S.P. 1,253,556 and 1,345,305–6; B., 1918, 174 A; 1920, 564 A), and then reflocculated by adding an electrolyte and more water, up to about 40%, while continuing the grinding. The resulting paste is filter-pressed, dried, and if necessary (*e.g.*, for use as an adsorbent) calcined. Reflocculated materials prepared as described have very large surface per unit weight and are of special value for use as fillers and adsorbents. R. B. CLARKE.

Annealing furnace. G. H. COLE, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. CO. (U.S.P. 1,569,356, 12.1.26. Appl., 8.8.22).—A number of refractory heat-insulating walls enclose an open-top furnace chamber. An inner cover of low thermal resistance and an outer cover of relatively high thermal resistance are sealed in members of channel section disposed at the top of the walls near the inner and outer edges respectively. M. COOK.

Refrigerant. R. R. STITT (U.S.P. 1,570,080, 19.1.26. Appl., 2.2.25).—A product for use as a refrigerant or for other similar purposes consists of methyl chloride and methyl alcohol. W. CLARK.

Filtering apparatus. P. H. CRAWFORD and J. F. ABBEMA (U.S.P. 1,570,374, 19.1.26. Appl., 31.3.21).—A number of independent filtering units are capable of rotation on a carrier and the carrier also rotates so as to dip them in succession into a trough containing the material to be filtered. B. M. VENABLES.

Refrigerating apparatus. W. A. MCNUTT, Assr. to KITSON ENGINEERING CO. (LONDON), LTD. (U.S.P. 1,571,633, 2.2.26. Appl., 12.12.23).—See E.P. 214,736; B., 1924, 543.

Preparation of colloid solutions. C. S. SMITH (E.P. 246,570, 28.10.24).—See U.S.P. 1,514,737; B., 1925, 991.

Closed furnace and method of operating it. C. STEENSTRUP, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,571,742, 2.2.26. Appl., 20.12.23).—See E.P. 226,559; B., 1925, 853.

Rotary furnace. A. H. PEHRSON, Assr. to A. P. PEHRSON (U.S.P. 1,572,805, 9.2.26. Appl., 15.6.25).—See E.P. 238,928; B., 1925, 867.

Centrifugal separator. F. W. MCENTIRE (U.S.P. 1,572,299, 9.2.26. Appl., 18.6.23).—See E.P. 225,396; B., 1925, 418.

Apparatus for heat treatment by means of a bath of molten metal. A. McD. DUCKHAM, Assr. to THERMAL INDUSTRIAL AND CHEMICAL (T.I.C.) RESEARCH CO., LTD. (U.S.P. 1,572,779, 9.2.26. Appl., 20.6.23).—See E.P. 198,625; B., 1923, 753 A.

Gas-fired furnaces. ASKANIA-WERKE A.-G. (E.P. 234,791, 24.4.25. Conv., 31.5.24).

Removal of residue from furnaces, retorts, or the like. M. SCHWABACH (E.P. 240,467, 23.9.25. Conv., 23.9.24).

Pulverulent fuel furnaces for water-tube boilers. KOHLENSCHEIDUNGS-GES. (E.P. 241,940, 23.10.25. Conv., 25.10.24).

Steam regenerative accumulators. W. MÜLLER and P. M. HEINRICH (E.P. 246,678, 17.2.25).

[Means for jarring collecting electrodes in] electrical precipitation apparatus. LODGE-COTTRELL, LTD., and N. STALLARD. From METALLBANK U. METALLURGISCHE GES. (E.P. 246,757, 23.9.25. Addn. to 176,713).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Coal ash and clean coal. R. LESSING (J. Soc. Arts, 1926, 74, 182—197, 205—218, 224—241; Fuel, 1926, 5, 17—23, 69—76, 117—124; cf. B., 1925. 33; J.S.C.I., 1925, 277 T, 345 T).—Recent work on the origin of the ash in coal is summarised. A study of the alumina : silica ratio shows that durain ash, in spite of its high aluminium content, cannot be derived from lycopods, but is practically identical in composition with clay, whilst the ashes of clarain and vitrain closely resemble plant ashes. The four constituents of banded bituminous coal from the same seam show remarkable variations in ash content; *e.g.*, the values for the Hamstead Thick Seam are :—fusain 15·59%, durain 6·26%, clarain 1·22%, vitrain 1·11%. Full analyses of the banded constituents from many coals prove that the ashes of clarain and vitrain represent the original plant ash, that the durain ash is derived mainly from clay, and that much of the ash in fusain comes from the carbonates or other salts of infiltered water. The bulk of the ash in commercial coal comes from the roof, floor, and dirt bands of the seam. The microstructure of the banded constituents throws some light on the relationship between them. It is suggested that the ash constituents will be found a useful index of the component parts of a coal seam. The chemical changes occurring during incineration are discussed in relation to ash analysis. Where a complete ash analysis is not made, a proximate analysis into water-soluble, acid-soluble, and acid-insoluble portions gives useful information. The methods of

determining the ash structure and distribution by examination of ash skeletons, and by X-ray photographs, are briefly described. Large blocks of massive coal can be disintegrated by the action of dilute acid on the cleat and partings. The proposed process for winning coal by the use of sulphur dioxide in this way is described. The attack on the cleat and partings by sulphuric acid produced by the oxidation of pyrites facilitates the disintegration of coals containing the latter, and thus has a bearing on the question of spontaneous combustion. Methods in use for the removal of ash from coal in preparing it for the market (coal washing and cleaning processes) are reviewed. In considering the influence of the mineral constituents on the utilisation of coal the following factors arise:—(1) the total amount of ash present; (2) catalytic activity of the ash in promoting the various chemical processes involved; (3) fusibility of the ash. From the point of view of amount only, the ash acts as a diluting impurity. The work of Fieldner and his collaborators on the fusibility of coal ash is discussed, and a distinction is drawn between the behaviour of the ash in the furnace and when finely ground and mixed for m.p. tests. The most important function of the mineral matter in the carbonisation and combustion of coal is its catalytic activity. The yield of coke from the carbonisation of sugar and cellulose is greatly influenced by addition of small quantities of inorganic salts. The yield and composition of coke from bituminous coals is similarly influenced. Catalysts that increase the coke yield also tend to raise the carbon percentage in the coke. The catalytic influence of the inorganic matter has a bearing also on the gasification of coal and coke in producers, and on the hydrogenation of coal by the Bergius process. Future developments in the treatment of raw coal will probably tend to a more complete elimination of ash before putting the coal on the market. A. B. Manning.

Initial decomposition of coal by heat. M. J. Burgess and R. V. Wheeler (Fuel, 1926, **5**, 65—68).—A Lancashire caking coal was heated *in vacuo* and the gaseous products evolved over different temperature ranges were examined separately. Up to 200° the gases were mostly paraffin hydrocarbons which had been occluded in the coal. Between 270° and 300° the nature of these gases underwent a change, oxides of carbon appearing in quantity. Traces of a reddish-brown oil appeared also. These observations are considered to point to a decomposition of the coal substance initiated at 270°—280°. The coal was further heated from 300° to 400° and the gaseous distillation products were fractionated by liquid air to separate the paraffin hydrocarbons, the diversity of which could not have been disclosed by the ordinary methods of gas analysis. The presence of members of the paraffin series up to pentane was demonstrated. R. A. A. Taylor.

Conditions underlying the caking of coal. E. Audibert (Compt. rend., 1926, **182**, 316—318).—The temperature of incipient distillation of bituminous coal lies somewhat below that of incipient fusion, which latter sets in at temperatures between 325° and 450°, whatever the method of heating. On subjecting samples of French coals containing 21—42% of volatile matter to sudden heating at various temperatures, definite fusion-temperatures lying between the above limits have been found. If, however, the heating is gradual, caking sets in at temperatures 25—35° higher, or else not at all; this is ascribed to loss of volatile matter. Coals such as are used for metallurgical coke require a rate of heating of at least 1° per min. to give caked masses. W. A. Caspari.

Influence of temperature on the coke yield in crucible tests. H. Strache and C. Mika (Gas- u. Wasserf., 1926, **69**, 105—107).—The specification of the height of flame and the position of the crucible in the volatile matter determination for coal is shown to be an insufficient control of temperature, and the actual temperature inside the crucible should be determined. The nature of the crucible (platinum, nickel, V_2A steel) only influences the rapidity of coking and not to any appreciable extent the final coke yield (cf. J.S.C.I., 1922, 372 T). The method recommended is to heat 1 g. of coal in a metal crucible of 20—35 mm. diam. with a well-fitting lid for 7 min. so that the interior of the crucible rapidly attains a temperature of 900° and is kept at that temperature during the test. A. C. Monkhouse.

Importance of combustibility of the coke in the combustion of solid fuels on the fire-grate. A new heat theory. D. J. W. Kreulen (Brennstoff-Chem., 1926, **7**, 54—57).—Korevaar's theory of "heat compression" ("Combustion in the gas producer and the blast furnace," 1925) is applied to the combustion of fuels on the fire-grate, and the following relation deduced between the height (h) of the combustion zone, and the average temperature (T):

$$h=[x(667-0{\cdot}24t-0{\cdot}228T)-(T-T_k)K_3]/(T-T_0)K_2$$

where x is the weight of air in kg. at temperature t, entering the layer of coke per minute, T_k is the temperature of the boiler plate, and T_0 is the average temperature of the place where the heat loss occurs. Thus, other conditions being constant, T rises when h decreases. Korevaar has shown that the height of the oxidation zone in the gas producer is dependent on the combustibility of the coke in such a way that an increase of combustibility decreases h. This concept of combustibility involves the four factors, particle size, porosity, activity, and ash content. From the point of view of its behaviour on the fire-grate, a fuel is fully characterised by its moisture content, ash content, and the yield and combustibility of its coke. The theory is used to explain the apparently abnormal formation of clinker with certain coals. A. B. Manning.

Detection of carbon monoxide in fire-damp, combustion gases, and after-damp. L. Wein (Glückauf, 1925, **61**, 1623—1625; Chem. Zentr., 1926, I., 1456).—0·02—0·03% of carbon monoxide

in fire-damp can be detected with certainty by 5% palladous chloride solution. The test fails for combustion gases containing more than 1% of hydrogen. Unsaturated hydrocarbons must first be removed by absorption in bromine. In testing the after-damp from explosives any hydrogen sulphide formed must also first be removed. A. B. MANNING.

Condensation and wet [gas] purification. T. V. BLAKE (Gas World, 1926, 84, 102—105).—The efficiency of the condensing plant of a Glover-West continuous vertical retort installation carbonising a mixture of English and Welsh coals was examined. 3·7% of the ammonia was removed in the cyclone tar extractor, 74·1% in the air- and water-cooled condensers, 8·1% in the Livesey washers, and 13·9% in the rotary brush washers. 9·3% of the tar fog was removed in the extractor, 18·3% in the condensers, 52·4% in the Livesey washer, and 16·9% in the rotary washers. The naphthalene, hydrogen sulphide, carbon dioxide, and cyanogen contents of the gas, together with the temperatures, and composition of the liquor and tar were also determined at the various points of the system.
A. C. MONKHOUSE.

Artificial petroleum from Balkash sapropelite. N. D. ZELINSKY (Brennstoff-Chem., 1925, 6, 365—369; 1926, 7, 35—37).—The oils obtained by the dry distillation of a sapropelite from Lake Balkash have been examined. The sapropelite consists of a dry, clastic mass of characteristic wax- or tallow-like odour, and is remarkable for its high content of organic matter. Analysis gave moisture 1·8%, ash 4%; for the organic matter: C 73·76%, H 10·91%, S 1·03%, N 0·56%, P trace, O 13·74%. Organic solvents extracted from 27% (ether) to 39% (carbon tetrachloride) of the organic matter, the extract consisting principally of free fatty acids and their esters (waxes). 4930 g. of the material were distilled in two stages, giving (*a*) 2248 g. of tar and 562 g. of liquor; (*b*) 307 g. of tar and 795 g. of coke. The first tar fraction was mobile, of an intense garnet-red colour, and had *d* 0·82. It was washed with dilute sulphuric acid, distilled with superheated steam, and the distillate washed with potassium hydroxide solution and fractionated, giving 383 g. of light oil boiling to 150°, 1228 g. b.p. 150° (760 mm.) to 200° (20 mm.), and 353 g. boiling above 200° (20 mm.). These products, which contained from 10 to 40% of unsaturated compounds, and the second tar fraction were refined by treatment with sulphuric acid, and again fractionated. The crude light oil fractions were stable, those boiling below 120° showing no change after being kept under ordinary conditions for several years. The refined fractions resembled closely in physical properties (odour, density, and refractive index) and chemical composition the corresponding fractions from natural petroleum. The refined light oil had a boiling range of 58° to 137°, d^{11} 0·717, and *n* 1·4071. It consisted principally of paraffin hydrocarbons from hexane to nonane, and contained also some aromatic hydrocarbons and naphthenes. In particular the presence of methyl*cyclo*hexane was proved, and that of derivatives of *cyclo*pentane shown to be probable. Crystalline solid paraffins separated from the higher fractions. These fractions exhibited weak optical activity. On treatment with anhydrous aluminium chloride they gave products of the same chemical character as those obtained from Russian petroleum by the action of the same reagent. The close similarity between the oils from the Balkash sapropelite and the natural petroleum fractions gives further experimental support to the hypothesis of the organic origin of petroleum. A. B. MANNING.

Hydrogenation and desulphurisation of Norfolk shale oil. H. G. SHATWELL (J. Inst. Petrol. Tech., 1925, 11, 548—555).—Crude shale oil having *d* 0·9850, sulphur content 6·81%, and iodine value (Wijs) 882, was submitted to Bergius treatment with hydrogen in a gas-heated cylindrical autoclave. The pressure rose to about 100 atm., and the temperature was maintained at about 400° for 3 hrs. A similar experiment was made in the presence of nitrogen, the pressure rising to about 150 atm. The loss in treatment with hydrogen was 10% and with nitrogen 14%. The final pressures in the two experiments were 4 atm. below and 3 atm. above the respective initial pressures. The products had *d* 0·9463 and 0·9712 respectively. They were refined with 10% of 10% caustic soda and 2% of 98% sulphuric acid, again treated with alkali, and washed with water. The refining losses for the hydrogenated product were 35·4% for the petrol fraction and 24·7% for the kerosene. The refined products darkened slowly on keeping. The refined petrol had a sulphur content of 2·78%, only reduced to 2·31% by a second refining with 6% loss. The refined kerosene had a sulphur content of 5·39%. The hydrogenation greatly increased the yield of lighter fractions (from 6% to 22·3% at 175°).
H. MOORE.

Action of sulphuric acid on cracked spirit. C. M. HOUGHTON and S. BOWMAN (J. Inst. Petrol. Tech., 1925, 11, 583—586).—The behaviour of motor spirit in an engine depends on the proportion of paraffin, aromatic, naphthenic, and unsaturated hydrocarbons present. To ascertain the strength of sulphuric acid most effective in polymerising olefines, 100 c.c. of spirit were distilled, the final b.p. and the amount of reflux into the flask on removing the flame being noted. Another sample was distilled up to the final b.p. of the original spirit after treatment with acid. The difference between the volume remaining and the reflux in the first case was taken as a measure of the polymerisation. The cracked spirit used was entirely soluble in 95% acid. A portion was diluted with an entirely saturated spirit, and the mixture had a final b.p. of 108°, whilst 2% of reflux collected. Its fraction up to 100° was shaken for 5 min. with 50 c.c. of acid of 95, 92·5, 90, 87·5, and 85% strength, respectively. A drop in the reduction in volume between 95 and 92·5% acid probably indicated the minimum strength for solution of aromatic hydrocarbons. A rise in

high-boiling fractions occurred at about 91%. Experiments using the entire original spirit mixed with saturated spirit and treated twice with acid gave similar results, and 91% seems to be the optimum strength of acid for producing polymerisation in spirit of this class. H. Moore.

Determination of unsaturated constituents in petroleum spirit. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1925, **11**, 533—536).—The formation of gummy substances is probably due to substances of the diene class and not to mono-olefines. Unsaturated constituents may be determined by subtracting the quantity of aromatic constituents present from the total loss on treatment with strong sulphuric acid (98%); by addition of the gum value to the loss with 80% acid; or by calculation from the bromine value and the molecular weight. The Hanus test for bromine absorption was used. Molecular weights were determined by the Victor Meyer method, allowance being made for the resinous residue left in the tube. Aromatic constituents were determined by the Tizard–Marshall method (J.S.C.I., 1921, 20 t), and acid loss by shaking 100 c.c. of spirit with 300 c.c. of acid and keeping overnight. In (i.) a mixture of No. 3 spirits, (ii.) a shale petrol, and (iii. and iv.) typical cracked spirits, the percentages of unsaturated constituents found were, by the first method, 3·5, 10·0, 5·4, and 9·6; by the second method, 2·1, 12·5, 5·1, and 8·1, and by the third method, 3·8, 37·8, 13·6, and 9·4, respectively. The results given by bromine absorption appear to be too high. H. Moore.

Composition of cracked spirit. H. Moore and R. B. Hobson (J. Inst. Petrol. Tech., 1925, **11**, 587—592).—Investigations were made of the composition of a cracked spirit, made from Russian kerosene in a Cross cracking unit, and slightly refined with acid and soda. It had *d* 0·748, initial b.p. 34°, and final b.p. 167°. It was divided into four fractions: up to 95°; from 95° to 122°; from 122° to 150°, and residue at 150°. The mean mol. wts. of the fractions were calculated to be 91, 105, 119, and 140. The iodine value of the spirit was 56·3, and the figure for the unsaturated content was 24·9%. Naphthenes were determined by the aniline cloud test method after treatment with 98% sulphuric acid, and found to be 47·7%. An attempt was made to remove olefines by washing with 80% sulphuric acid. The product after washing gave an iodine value of 25·3. It was found that equilibrium was reached when 55% of the olefines had been removed. Further experiments showed that 90% acid was necessary to remove the olefines effectively. Assuming that no aromatic hydrocarbons had been removed, the aromatic hydrocarbons were responsible for lowering the aniline point 3·9°, corresponding to an aromatic content of 4·7%. Further experiments indicated that 90% acid removed the unsaturated hydrocarbons almost completely, and 1·1% of the aromatics. By separate analysis of the fractions there were found 22·5% of unsaturated hydrocarbons, 50% of naphthenes, 6·8% of aromatics, and 19·6% of paraffins, a result not greatly differing from that obtained by the other methods. The results obtained are very different from those of Ormandy and Craven (cf. preceding abstract). H. Moore.

Determination of the molecular weight of petrol. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1925, **11**, 539—542).—In determinations by the Victor Meyer method erratic results were obtained at temperatures above 220°. This was particularly the case with heptane, but better results were obtained when fragments of porous earthenware were introduced into the tube. With benzene no irregularity was observed. This effect seems to be due in part to cracking, but also to actual combustion of the hydrocarbon. Cracking action does not seem to increase with rise of temperature above the critical, and mostly takes place in the liquid state. Better results are obtained by replacing the air in the tube by coal gas or carbon dioxide. The molecular weight of a petrol was found by the Victor Meyer method to be 100—101, and by the freezing-point method to be 118. The formation of carbon dioxide in mixed heptane vapour and oxygen passed over heated iron begins at 241°, and with toluene and oxygen at 210°. When petrol was heated in an atmosphere of its own vapour no cracking was observed up to 500°. H. Moore.

Valuation of motor spirit and lubricating oils. H. Moore (J. Inst. Petrol. Tech., 1925, **11**, 571—575).—The tests essential for the valuation of lubricants are specific gravity, viscosity, cold test, flash point, coke test, and appearance. The gravity, in connexion with other values, may indicate the origin of the oil; high flash point indicates close fractionation in refining. The readings of viscosimeters in general use are not direct functions of absolute viscosity. Load factor and age of engine may decide the viscosity of oil to be used, but the size of the engine alone is not determinative. Pennsylvanian oils fall off less in viscosity with rise in temperature than do Texas and Californian oils. The heavier blends of cylinder oils contain at least two different bases. The presence of paraffin wax leads to high cold-test results. Wax-free oils may be obtained with cold tests below −7°. The coke test indicates liability to carbon deposit in cylinder, engine conditions being the same. In the same class, the lighter coloured oil is generally the better. Compounded oils have the faculty of maintaining a film under extreme pressure. The tests for motor spirit are specific gravity, Engler distillation, corrosion test, and aniline cloud test. The higher the specific gravity the greater the power obtainable per unit volume. Ease of starting is determined by the amount of spirit distilling over from initial b.p. to 100°. The aniline cloud test is a measure of the aromatic hydrocarbons present, and of the tendency to detonate. Lead tetraethyl as an anti-detonator is objectionable because of its poisonous qualities. Ethyl alcohol may come to be used for this purpose. H. Moore.

Adsorption and electrical properties of various charcoals. Ogawa.—See A., Mar., 239.

PATENTS.

Peat drying machine. C. A. WILLMARTH (U.S.P. 1,569,345, 12.1.26. Appl., 16.11.23).—Horizontal drums open at the ends and provided with screw conveyors are arranged in two rows, an upper and a lower, within a heating chamber, and project through the walls of the chamber into compartment heads common to all the drums. One of the heads has an inlet opening, the other a discharge opening.
R. A. A. TAYLOR.

Burning fuel. FULLER FUEL CO., and G. H. KAEMMERLING (E.P. 224,511, 4.10.24. Conv., 6.11.23).—The system is designed essentially for effecting in small space the complete combustion of gaseous, liquid, and, in particular, pulverised fuels and consists of a combustion chamber with a number of burners pointing through holes in the wall tangentially directed to an imaginary circle in the middle, the jet from each burner impinging on that from the burner before it. The result is a rotating zone of combustion in the chamber, which is well-shaped with respect to a circulating chamber. Various modifications are described and provision is made for cooling the combustion chamber walls by air or water. R. A. A. TAYLOR.

Preparation of combustible and heating materials from peat and the like. CHEM. FABR. HEPPES U. CO., and J. B. CARPZOW (G.P. 421,734, 24.3.23).—In preparing fuels from humic substances by converting them into coal-like substances by biological processes under the action of bacteria and fungi, fresh sea or river-slime (sapropelic mud) may be mixed with the material. The mixture is left in pits to mature, then dried, and pressed.
R. A. A. TAYLOR.

Vertical retorts for the distillation of shale and like materials. R. H. CROZIER (E.P. 245,499, 7.10.24).—A retort is built up of flat standard sections, formed with flanges so that they may be bolted together, and semi-cylindrical end sections, the opposite flat sections being connected by bracing members in the form of oval internal flues. The retort may be extended and its capacity increased by detaching the sections at one end, attaching additional flat sections, and re-attaching the end sections. The internal bracing flues may be provided with means for withdrawing the vapours from the retort, and for supplying steam thereto. A. B. MANNING.

[Retort for] carbonising fuels. J. PLASSMANN (E.P. 240,800, 17.8.25. Conv., 30.9.24).*—A carbonising retort consists of superposed flat annular chambers, *a*, heated both above and below from the interior. The stationary superposed chambers are enclosed by a rotary jacket, *c*, to which are attached the supplying and discharging means. Bands, *h*, also attached to the outer jacket, extend into the carbonising chambers, and prevent the fuel from falling out, while allowing the passage of gases and vapours. The fuel is supplied to the chambers from a central bunker above the retort under the pressure of the screw feeds, *k*, whereby a particularly dense semi-coke is obtained. The coke is removed by a rotating scraper, *n*, and is quenched in such a way as to retain sufficient heat to evaporate the

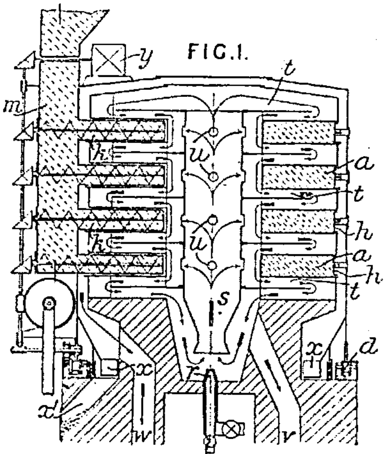


FIG. 1.

FIG. 3.

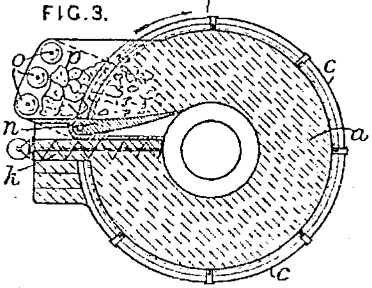
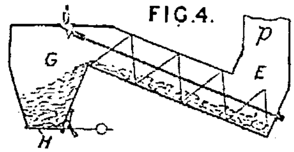


FIG. 4.

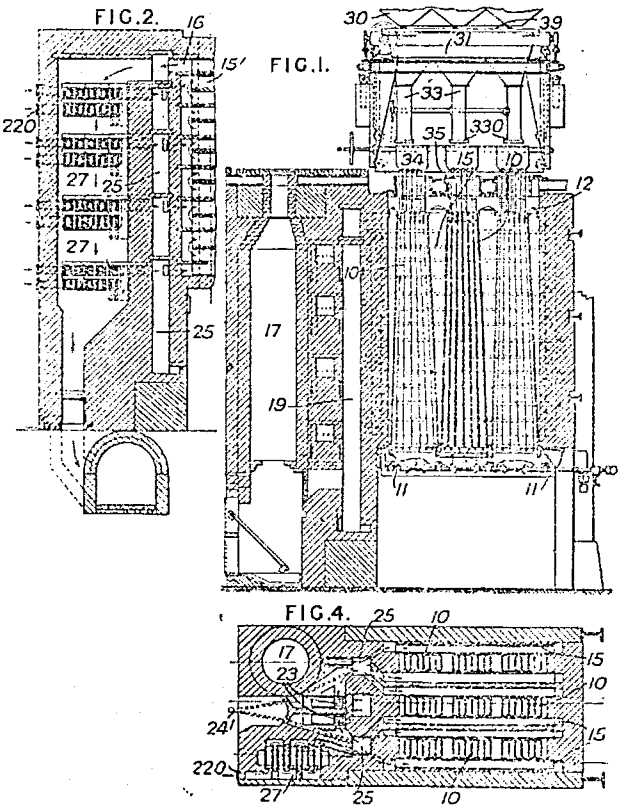


water adhering to the coke. For example, it may be discharged into an inclined conduit, *e* (Fig. 4), containing water, and conveyed thence into a collecting chamber, *g*. The chambers are heated by a central heating cell, provided with a burner, *r*, the heating gases being led around the chambers by the annular guide discs, *t*. The gases and vapours formed are removed by suction through the channel, *w*. A. B. MANNING.

Manufacture of active charcoal. P. M. A. LEBEAU (F.P. 595,400, 18.6.24).—The substance to be carbonised, *e.g.* wood, is cut into the form of cubes, prisms, cylinders, etc. These are impregnated by prolonged immersion in a solution containing dilute sulphuric acid and a metal sulphate, *e.g.* aluminium sulphate, chromium sulphate, etc. Carbonisation is effected in two stages. The temperature is slowly raised to 500—600° to avoid sudden loss of volatile matter and shattering of the blocks. It is then raised to 600—900°; during this stage the metal sulphate is reduced and carbonisation completed R. B. CLARKE.

Carbonisation of fuel. S. R. ILLINGWORTH, ILLINGWORTH CARBONIZATION CO., LTD., R. DEMPS-

ter and Sons, Ltd., and H. J. Toogood (E.P. 245,190, 25.9.24).*—The setting consists of a producer, 17, and cast iron vertical retorts, 10, made



of tapering tubes, 2 in.—7 in. diam., or of plates of H-section, each tube being a separate unit and easily removable. The retorts are enclosed by horizontal fireclay flues, 15, which are so arranged that the heat can be varied at different levels, and a chamber for cooling the fuel or for further carbonisation is provided below the retorts. The setting is particularly suitable for the manufacture of shaped fuel from blended or non-swelling coals.
A. C. Monkhouse.

Conversion of poorly coking coals into useful metallurgical coke. H. Koppers (G.P. 415,846, 31.8.22).—A finely ground mixture of a poorly coking coal and a more gassy coal is briquetted and from the briquettes a product similar to the coal cakes obtained by the usual method of tamping, but higher and narrower, is prepared. This is charged into horizontal ovens with narrow high chambers.
R. A. A. Taylor.

Operation of carbonising and gasifying plant. P. Illig (G.P. 416,540, 21.4.23).—In plant where the gasifying portion is separated from the carbonising retort by an intermediate chamber provided with a conveyor, the passage of the charge from the retort is so regulated that only carbonised material in a loose (divided) condition enters the gasifying chamber, and sufficient room is left in the intermediate chamber to admit of cooling the gas passing from the gasifying chamber to the retort by any desired means such as added gas, steam, or a built-in heat exchanger.
R. A. A. Taylor.

Carbonisation of bituminous substances. F. Leitner (G.P. 417,211, 24.10.23).—The charge is carbonised in an externally heated continuous inclined or vertical retort, and gas or a mixture of steam and gas heated by sensible heat derived from the spent charge is introduced into the retort. By suitable means the charge in the retort is subdivided into numerous small portions, so that the gas or gas–steam mixture is deflected alternately from the retort wall to the charge and *vice versa.*
R. A. A. Taylor.

Vertical retort for low-temperature carbonisation. W. Pfeiffer (G.P. 420,810, 31.12.22).—The fuel passes through a container consisting of co-axial vertical cylinders connected by screw-blades; the fuel does not fill the space at its disposal. The retort moves to and fro about its vertical axis, and the heating gases circulate in the annular space between the two cylinders. The screw-blades are provided with steps or weirs to ensure mixing and turning over of the fuel. Inside the inner cylinder is a collector with dust-separator for the distillation gases.
R. A. A. Taylor.

Carbonisation process for wood, peat, shale, and the like. Norddeutsche Torfkokerei A.-G. (G.P. 421,119, 21.10.24).—In intermittent vertical retorts where the charge is admitted a layer at a time, hot gases are admitted above and pass downwards long enough to bring each fresh layer to about 200°, when they are cut off until a fresh layer has been introduced. R. A. A. Taylor.

Cooling coke. J. Y. Johnson. From Badische Anilin- u. Soda-Fabr. (E.P. 245,702, 15.10.25).—Coke is cooled, and at the same time a useful fuel gas produced, by passing carbon dioxide, or a gas rich in carbon dioxide, through it. The sensible heat of the resulting gas may be utilised prior to its combustion, *e.g.*, by passing it through the tubes of a steam boiler.
A. B. Manning.

Manufacture of water-gas. Humphreys and Glasgow, Ltd., Assees. of O. B. Evans (E.P. 231,866, 31.3.25. Conv., 3.4.24).—The plant consists of one or two generators situated between two sets of carburettor and superheater. The air or steam, superheated in one set, enters the first generator at the middle of the fuel bed and the gases leave at the top and bottom; they then enter the second generator at the top and bottom and leave at the middle to heat up the second carburettor and superheater. In the succeeding "blow" and "run" the direction of flow is reversed. The hot gases do not pass through the grates, and if coal

be used provision is made for the introduction of secondary air at the top of the fuel bed.
A. C. Monkhouse.

Gas producer. S. A. Moss, Assr. to Gen. Electric Co. (U.S.P., 1,570,314, 19.1.26. Appl., 27.2.23).—The air–steam blast for the producer is supplied by means of a steam jet, the area of the throat of the discharge tube being not more than 100 times greater than the steam nozzle throat area.
A. C. Monkhouse.

Removal of sulphuretted hydrogen from gas. Woodall-Duckham, Ltd., E. W. Smith, and T. C. Finlayson (E.P. 245,575, 10.12.24).—Gas to be purified is washed in a tower scrubber with a solution of alkali containing a suspension of iron compounds. The spent liquor withdrawn from the bottom of the tower is regenerated by repeated emulsification with air, supplied under such a pressure as to lift the liquor during this process into one compartment of the tank at the top of the tower. Sulphur which is formed during regeneration rises as a froth to the surface of the regenerated liquor and overflows into a centrifuge, where it is separated. If necessary the liquor may be treated for regeneration more than once and then utilised for the purification of more foul gas.
S. Pexton.

Gas washer and scrubber. J. F. Blakeley (E.P. 245,814, 12.9.24).—The scrubber is of the horizontal type and consists of chambers in which rotate a number of circular brushes on separate shafts, the direction of rotation of which can be automatically reversed. The gas enters a preliminary chamber through a strainer and comb dipping into the liquid in order to remove the heavier constituents of the gas before passing to the brushing chambers.
A. C. Monkhouse.

Purification of acetylene. A. Wacker, Ges. für Elektrochem. Ind., Assees. of W. Gruber (G.P. 419,729, 20.9.24).—The gas, with small quantities of oxygen, or a gas containing oxygen, is passed over silica gel at elevated temperatures. The phosphorus and sulphur compounds present as impurities are oxidised, and the oxidation products can easily be removed from the gel by water or steam.
R. A. A. Taylor.

Increasing the safety and activity of porous masses for the storage of acetylene. Autogen Gasaccumulator A.-G., Assees. of E. Steil (G.P. 420,150, 2.11.24).—Organic substances used as porous masses for storing explosive gases such as acetylene may be treated, before or after insertion into the container, with calcium chloride and water-glass or with solutions of zinc or copper salts and resin soap, whereby finely-divided inorganic matter is deposited on the porous material, rendering the organic substance non-inflammable and decreasing the size of its pores, thus enhancing capillary action.
R. A. A. Taylor.

Distillation of [benzol] wash-oil. Zeche M. Stinnes, Assees. of A. Weindel and H. Kiemstedt (G.P. 419,570, 7.2.24).—To increase light oil yields, the first runnings and some of the benzol are collected as a first fraction and are passed to a washer situated between the condenser and receiver and fed by the condensate from the receiver.
R. A. A. Taylor.

Fuel. G. Hammond, Assr. to Fuel Development Corp. (U.S.P. 1,570,059—60, 19.1.26. Appl., [a] 17.6.22, [b] 3.5.23).—Combustible mixtures consisting of (a) kerosene, a light readily ignitible hydrocarbon, and small quantities of acetone, glycerin, and butyl alcohol; or (b) kerosene, gasoline, butyl alcohol, and benzol, are claimed.
W. Clark.

Motor fuel. R. H. McKee (U.S.P. 1,570,161, 19.1.26. Appl., 19.11.23).—A homogeneous mixture of the gasoline-like fraction of shale-oil, alcohol, and ethylene.
L. M. Clark.

Purification of hydrocarbons. T. T. Gray (E.P. 222,481, 23.9.24. Conv., 26.9.23).—The vapours from a still in which heavy oil is distilled or cracked are passed around a vessel containing a catalyst, which may be fuller's earth, bone-black, bauxite, kieselguhr, etc., so as to raise the catalyst to the temperature of the vapours. The vapours then pass through the catalyst, when certain of their unsaturated constituents are polymerised to form liquid compounds of higher boiling point. The reaction may be exothermic, and the products may be returned to the still or collected. The product obtained by condensation of the vapours is improved in quality, odour, and colour, and is superior to that obtained by sulphuric acid refining. The catalyst remains at a temperature below the b.p. of the polymerides.
H. Moore.

Vapour separation and condensation particularly for use with [petroleum] oils. Power Specialty Co. (E.P. 239,208, 25.8.25. Conv., 27.8.24).—The vapours are treated in a number of rectifying columns at successively lower temperatures and are finally exhausted to a condenser, part of the liquid from the condenser being used as reflux liquor in the final column. The condensate formed at any stage is passed through pipe coils within the lower parts or condensate sumps of all later stages and serves to re-distil the later condensates, and each condensate after passing through a final cooler is partly returned as reflux liquor to the next earlier stage.
B. M. Venables.

Cracking hydrocarbon oils. C. P. Dubbs, Assr. to Universal Oil Products Co. (U.S.P. 1,568,400, 5.1.26. Appl., 4.2.25).—The oil is passed from an elongated heating coil in a furnace to an enlarged reaction zone, and thence the vapours pass to a dephlegmator. Unvaporised oil may be withdrawn from the reaction zone without re-entering the heating coil. Reflux oil from the dephlegmator may be pumped into the heating coil under pressure, or may be admitted continuously to the coil without pumping.
H. Moore.

Treatment of low-boiling hydrocarbons. W. F. DOWNS (U.S.P. 1,568,812, 5.1.26. Appl., 6.5.22).—The yield of gasoline is increased and its quality improved by condensing the vapours of hydrocarbon oils which boil below 260° in a porous mass containing anhydrous metal chlorides. The liquid condensate is continuously withdrawn from contact with the chloride. H. MOORE.

Purifying hydrocarbon liquids. W. F. DOWNS (U.S.P. 1,568,813, 5.1.26. Appl., 6.5.22).—Hydrocarbons are passed in liquid form through a filter bed containing a metal chloride heated to a temperature below the b.p. of the hydrocarbon and below the vaporising point of the chloride. H. MOORE.

Refining hydrocarbon oils. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,568,904, 5.1.26. Appl., 27.4.23).—The oil is subjected separately to the action of sulphuric acid and of caustic soda containing litharge, sulphur dioxide being afterwards added. H. MOORE.

Treating [hydrocarbon] oil. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,569,855, 19.1.26. Appl., 20.8.20).—Vapours generated in a still for treating hydrocarbon oils, pass through a filter bed situated in the vapour space above the oil level, and above the oil inlet into the still, and then into a dephlegmator. Vapour leaving the dephlegmator passes to a condenser, whilst condensed liquid is returned to the still through an opening situated below the filter bed. L. A. COLES.

Refining [mineral] oils. Refining hydrocarbon oil. Refining cracked hydrocarbon oil. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,569,870–2, 19.1.26. Appl., [A] 28.8, [B, C] 31.8.22).—Oil is treated with alkali plumbite solution, and after removing the solution as completely as possible, is treated with (A) a metal sulphide capable of hydrolysing in water to complete the reaction of the plumbite solution, or (C) an aqueous sulphide solution, precipitated material and sulphides being subsequently removed. (B) Oil is treated successively with sodium plumbite solution, with an aqueous sulphide solution, and with a flocculating agent to precipitate the other products of reaction, the aqueous solutions and precipitated material being removed after each stage. L. A. COLES.

Medium for bleaching, cracking, and desulphurising petroleum and other hydrocarbon compounds. H. and H. REINBOLD (U.S.P. 1,570,005, 19.1.26. Appl., 21.1.24).—Oil is treated with hydrated silicic acid in combination with a metal chloride. L. A. COLES.

Treating [cracking] petroleum oils. R. B. DAY (U.S.P. 1,570,131, 19.1.26. Appl., 5.8.21).—High-boiling hydrocarbon oil is partially cracked by heating in a chamber connected with an upper chamber by means of a pipe extending from the top of the lower to the bottom of the upper chamber. The lower chamber and the pipe are filled, and the upper chamber is partly filled, with the oil under treatment. Vapours generated during the treatment are withdrawn from the upper chamber. Deposition of carbon on the walls of the heating chamber is prevented by continuously agitating the oil therein. L. A. COLES.

Desulphurisation of oils, hydrocarbons, and tars. A. A. M. MASSENET (G.P. 421,263, 28.6.22).—The liquid is intimately mixed with and distilled over a small quantity of desulphurising material, such as a metal, metallic compound, alloy, or amalgam, *e.g.*, a 50% emulsion of a metal, such as sodium, with oil, tar, or petroleum. The emulsion may be prepared from finely-powdered or molten metal by shaking at elevated temperature with a hydrocarbon, preferably one which is solid at ordinary temperatures, such as naphthalene or transformer oil or a fatty oil. R. A. A. TAYLOR.

Improvement of cracked distillates. OBERSCHLESISCHE KOKSWERKE U. CHEM. FABR. A.-G., F. RUSSIG and A. SUPAN (G.P. 421,558, 1.1.22).—The crude products are treated with a moderate quantity of sulphuric acid (*d* 1·53), whereby the evil-smelling constituents are converted into substances with an aromatic odour. The products are separated by distillation into light products similar to benzene and a residual oil, the latter being returned to the cracking process. R. A. A. TAYLOR.

Preparation of a catalyst for recovering light hydrocarbons from heavy mineral oils and shale oils. G. GRISARD (Swiss P. 111,354, 30.7.24).—Aluminium chloride is mixed with an alkali or alkaline-earth, a silicate, and a mineral oil having a high content of sulphur and unsaturated hydrocarbons, to give a pitchy mass, which is but slightly hygroscopic, and can be transported and stored. The product is used as a catalyst for cracking oils, being mixed with the latter by heating and stirring. R. A. A. TAYLOR.

Emulsion. J. C. MORRELL (U.S.P. 1,556,005, 6.10.25. Appl., 18.5.23).—By mixing together emulsions having as internal phase liquids of different viscosities, partially or completely miscible with one another, an emulsion having an internal phase of intermediate viscosity is obtained. For example an emulsion of a very viscous asphalt can be made more fluid by adding an emulsion of a creosote oil. T. S. WHEELER.

Product derived from acid sludge. H. T. BENNETT, G. B. MURPHY, and LE R. G. STORY (U.S.P. 1,568,261, 5.1.26. Appl., 20.12.24).—Acid sludge containing asphaltic substances is converted into useful products by mixing it with a solvent containing alcohol and a coal-tar product, *e.g.*, benzol, and distilling off the excess solvent. H. MOORE.

Recovery of aluminium chloride [from oil residues]. A. McD. MCAFEE, Assr. to GULF REFINING Co. (U.S.P. 1,568,171, 5.1.26. Appl., 25.1.22).—To recover aluminium chloride and oil, oily residues

are heated to a temperature sufficient to volatilise the chloride, under a minimal partial pressure of aluminium chloride vapour. H. MOORE.

Reclaiming and converting asphaltum and other materials from waste material [with production of lampblack]. R. D. DIVINE (U.S.P. 1,569,462, 12.1.26. Appl., 18.1.24).—The material is heated in an inclined retort, and melted material (asphalt) drops from the lower end of the retort on to a hot plate contained in a chamber connected with a lampblack collection chamber, the latter also being connected with the upper gas-discharge end of the retort. J. S. G. THOMAS.

Dust-like fuel. F. W. BRANDES (U.S.P. 1,571,621, 2.2.26. Appl., 3.7.24).—See E.P. 215,771; B., 1925, 793.

Method of making fuel briquettes. P. E. WELTON (U.S.P. 1,572,629, 9.2.26. Appl., 19.5.24).—See E.P. 244,971; B., 1926, 182.

Container for testing coal and method of testing. W. J. KLAIBER, Assr. to KOPPERS Co. (U.S.P. 1,572,391, 9.2.26. Appl., 12.9.23).—See E.P. 221,778; B., 1925, 390.

Apparatus for carbonising bituminous fuels. A. BESTA (U.S.P. 1,571,927, 9.2.26. Appl., 27.12.24).—See E.P. 226,809; B., 1925, 700.

Gas producer. J. F. ROGERS, Assr. to WELLMAN-SEAVER-MORGAN Co. (U.S.P. 1,572,041, 9.2.26. Appl., 12.11.21. Renewed 31.3.24).—See E.P. 188,669; B., 1923, 1213 A.

Purification of gas. J. W. COBB and H. J. HODSMAN (U.S.P. 1,571,624, 2.2.26. Appl., 5.1.25).—See E.P. 233,011; B., 1925, 533.

Fuel for internal-combustion engines. B. H. MORGAN (U.S.P. 1,572,750, 9.2.26. Appl., 4.4.25).—See E.P. 225,685; B., 1925, 91.

Heat-exchange device [for fractionating hydrocarbons]. H. L. ALLAN (U.S.P. 1,572,374, 9.2.26. Appl., 15.12.19).—See E.P. 152,791; B., 1920, 813 A.

Apparatus for production of gasoline. F. TINKER (U.S.P. 1,572,626, 9.2.26. Appl., 7.6.23).—See E.P. 205,289; B., 1923, 1213 A.

Apparatus for removing water from coal after washing. BUTTERLEY CO., LTD., and G. F. WRIGHT (E.P. 247,012, 10.2.25).

See also pages 236, **Ammonia** (F.P. 594,712); **Hydrogen–nitrogen mixture** (G.P. 419,860); **Ammoniacal liquor** (G.P. 420,498). 237, **Vanadium compounds from petroleum** (U.S.P. 1,570,170); **Carbides** (G.P. 421,268).

III.—TAR AND TAR PRODUCTS.

Manufacture of prepared tar for road-surfacing. C. AB-DER-HALDEN (Chim. et Ind., 1926, **15**, 14—17).—The binding qualities of road tar are injured by the presence of water-soluble constituents (phenols) and of volatile constituents (naphthalene). A continuous tar-dehydration plant is described in which distillation is effected in steam at 180° under reduced pressure. The tar produced contains 0·05% of water, 1·5% of naphthalene, and 0·03% of phenols, the distillate being separated into two fractions in a rectifying column. C. IRWIN.

Normal composition of the light oil from the low-temperature tar from bituminous coals. H. BROCHE (Brennstoff-Chem., 1926, **7**, 37—39).—The benzol and phenol contents of the low-temperature tar produced from Hardenberg coal have been determined (0·5% and 0·16% respectively), and shown to conform with the values generally accepted as characteristic of a low-temperature tar. The tar from the same coal, investigated by Schütz (B., 1923, 175 A, 488 A, 595 A, 1119 A; 1924, 367, 460), who found considerable quantities of aromatic hydrocarbons in the neutral oil boiling below 200°, and a phenol content of 1·35%, cannot have been a true low-temperature tar, but must have undergone some cracking. This conclusion is supported by other recent work on low-temperature tars (Klein, B., 1926, 148; Brittain, Rowe, and Sinnatt, B., 1925, 795). A. B. MANNING.

Free sulphur in benzol. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1925, **11**, 556—559).—To determine free sulphur 100 c.c. of benzol are shaken with 1·2 c.c. of mercury in a tap funnel, and a small quantity of dilute hydrochloric acid is added. The mixture is filtered under vacuum through a Gooch crucible with double filter paper at the bottom. The crucible with the precipitate is placed in a beaker and covered with *aqua regia*, and boiled until no mercury globules are left. It is then washed with water, the solution is neutralised with ammonia, 1 c.c. of hydrochloric acid added, and the liquid made up to 350 c.c. After filtering, barium nitrate is added to the filtrate, which is boiled and filtered, sulphur being determined as barium sulphate. The results are reasonably accurate. H. MOORE.

PATENTS.

Recovery of phenols and pyridine bases from tar distillation products or other mixtures. P. PAPILLON (F.P. 594,687, 30.5.24).—Before the usual fractional distillation, tar oils are washed at a little above 30° with alkali, the alkali extract then being run off and treated to recover the phenols. The residue is fractionally distilled, and the 90—160° fraction, which contains the greater part of the bases, is extracted with dilute sulphuric acid and the extract worked up in the usual way.
R. A. A. TAYLOR.

Distillation of benzol wash-oil (G.P. 419,570).—See II.

Desulphurising tars (G.P. 421,263).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Solubility of the nitroanilines in various liquids. COLLETT and JOHNSTON.—See A., Mar., 237.

Passivity of zinc dust in alkaline reduction of nitro-compounds. Izmailski and Kolpenski. —See A., Mar., 248.

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Manufacture of quinizarin. H. Dodd, W. C. Sprent, and United Alkali Co., Ltd. (E.P. 245,584, 29.12.24).—A product, which is substantially quinizarin, is obtained by heating *o*-chlorophenol or a mixture of *o*- and *p*-chlorophenols (crude monochlorophenol) with phthalic anhydride in presence of sulphuric and boric acids to temperatures above 200°. For example, 80 g. of *o*-chlorophenol in 150 g. of sulphuric acid (preferably monohydrate) are heated at 90—100° for 1 hr. A mixture of 120 g. of phthalic anhydride and 50 g. of boric acid in 750 g. of sulphuric acid is run into the first mixture with slow agitation. The mixture is slowly raised to about 250° during 16 hrs., cooled to 40—50°, and then run into 1500 c.c. of cold water. The quinizarin (about 88% yield) separates as a reddish-brown powder identical with that from pure *p*-chlorophenol. A. Coulthard.

Sulphonated [hydr]oxydianthraquinonylamine dyestuffs. W. Mieg and H. Raeder, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,564,091, 1.12.25. Appl., 17.8.25).—The products obtained by sulphonating dianthraquinonylamines as described in U.S.P. 814,137 (cf. F.P. 357,138, B., 1906, 66) are mixtures of sulphonic acids and hydroxysulphonic acids of the amines. According to the present patent by controlling the conditions of sulphonation products consisting entirely of hydroxysulphonic acids are obtained which are superior as dyestuffs to those obtained previously. For example, 1:1'- or 1:2'-dianthraquinonylamine is treated with oleum containing 15—30% SO_3 at 120—150° in presence or absence of boric oxide or mercuric oxide or other oxygen carriers. The resulting hydroxydianthraquinonylaminesulphonic acids dye wool from an acid bath brownish-violet to blue shades. With after-chroming, or if chrome-mordanted wool is used, brownish-violet to bluish-black shades are produced. The dyestuff from 1:1'-dianthraquinonylamine is converted into 1:1'-dianthraquinonyloxazine when heated to 140° with 95% sulphuric acid. T. S. Wheeler.

Manufacture of azo-dyestuffs. J. Y. Johnson. From Badische Anilin- u. Soda-Fabr. (E.P. 245,674, 13.7.25).—Azo dyestuffs of good solubility in water, which give yellow shades on both animal and vegetable fibres, can be obtained by the introduction of one or more sulphonic acid groups into disazo dyestuffs derived from diaminodiarylcarbamides and 2 mols. of salicylic acid or one of its derivatives. In order to introduce the sulphonic groups the disazo dyestuffs may be directly sulphonated, or the tetrazotised sulphonic acids of diaminodiarylcarbamides may be combined with salicylic acid derivatives, or the aminoazo dyestuff, aminoarylenesulphonic acid-azosalicylic acid compound, may be treated with phosgene. For example, 10 pts. by weight of the disazo dyestuff from *pp'*-diaminodiphenylcarbamide and two mols. of salicylic acid, are added at room temperature to 100 pts. of oleum (23%) while stirring. After 2 hrs. at about 75° the mass is poured on ice and the dyestuff filtered off and purified. A. Coulthard.

Manufacture of intermediate compounds and of azo dyestuffs. British Dyestuffs Corp., Ltd., and K. H. Saunders (E.P. 245,865, 21.10.24). —Aromatic *o*-hydroxycarboxysulphochlorides, such as salicylic acid sulphochloride, *o*-cresotic acid sulphochloride, and 3-chlorosalicylic acid sulphochloride, are converted by treatment with reducing agents other than alkaline sodium sulphite into the corresponding *sulphinic acids*, and these are condensed with aromatic nitro-compounds having a labile halogen atom, such as 4-chloro-3-nitrobenzenesulphonic acid, 4-chloro-3-nitrobenzenecarboxylic acid, 4-chloro-3-nitrobenzaldehyde, 2:4-dinitrochlorobenzene, 3:5-dinitro-4-chlorobenzenesulphonic acid, and 1-chloro-2:4-dinitronaphthalene, yielding *nitrosulphones*. The *aminosulphones* obtained by reducing the nitrosulphones, on diazotisation and coupling with azo components, yield *dyestuffs* having the chrome-fixing group screened from the chromophore group, so that very little change in shade takes place on mordanting. Disazo or polyazo dyestuffs may be produced, *e.g.*, by coupling with azo components containing amino-groups, and subsequently diazotising these and coupling with further azo components. For example, 2·8—3 pts. of zinc dust are added in small quantities, to prevent too great a rise in temperature, to a solution in 20 pts. of alcohol of 10 pts. of salicylic acid sulphochloride, which is obtained as described in E.P. 18,430 of 1913 (cf. G.P. 264,786; B. 1913, 1003). After removing the solvent by filtration, the residue is dissolved in strong sodium hydroxide and the *sulphinic acid* is precipitated by slowly adding acid until the solution is acid to Congo Red. A mixture of 10 pts. of the sulphinic acid, 13 pts. of sodium 4-chloro-3-nitrobenzenesulphonate, 5·3 pts. of sodium carbonate, and 25 pts. of water, is boiled for several hours, and the *nitrosulphone*, $(1)OH\cdot(2)COOH\cdot C_6H_3\cdot(4)SO_2\cdot C_6H_3\cdot(2)NO_2\cdot(4)SO_3H$, is precipitated by acidifying the hot solution, or is converted into its barium salt, or is reduced direct to the corresponding *aminosulphone*, *e.g.*, by the addition of iron filings or zinc dust and an acid, or stannous chloride or sodium sulphide, or it may be chlorinated by treatment with an alkali hypochlorite before reduction. The amine on diazotisation and coupling with β-hydroxynaphthoic anilide, β-naphthol, β-naphthylamine, ethyl-β-naphthylamine, phenylmethylpyrazolone, and ethylbenzylaniline, yields dyestuffs giving salmon-pink, orange, scarlet, bluish-red, yellow, and orange shades, respectively, on printing with a chrome mordant on cotton. L. A. Coles.

Azo dyes containing chromium. Badische Anilin- u. Soda-Fabr., Assees. of H. Kämmerer (G.P. 419,825, 31.7.23).—Azo dyes capable of

chroming are treated first with chromium salts and then with alkaline substances. The chromium compounds obtained in this way can be separated from unused chromium salts by salting out. They dye wool and silk in fast shades from an acid bath. A. COULTHARD.

o-Hydroxyazo dyes. GES. FÜR CHEM. IND. IN BASEL (Swiss P. 107,204 and 107,520, 8.11.22).—Double compounds of an azo dye and β-naphthylamine are obtained by the action of 1 mol. of the diazo compound from 2-aminophenol-4-sulphonic acid or from 4-chloro-2-aminophenol-5-sulphonic acid, on 2 mols. of β-naphthylamine. By the action of sodium hydroxide, or other hydrolysing substance, β-naphthylamine is split off, leaving solutions from which, after filtration, the dyes may be salted out. They give orange shades on wool from an acid bath and can be used for the preparation of other dyes. A. COULTHARD.

Preparation of azo dyes. GES. FUR CHEM. IND. IN BASEL (Swiss P. 111,123 and 111,497—9, 12.4.24).—Fast bordeaux and black dyes are obtained by combining 2:4:6-tri-p-hydroxynaphthyl-1:3:5-triazine with the diazo compounds from 4-chloro-2-aminodiphenyl ether, aminoazobenzene, 4-amino-2′-ethoxyphenylazonaphthalene, or the tetrazo compound from 4:4′-diamino-5-methoxy-2-methylazobenzene, either in solution or on the fibre. A. COULTHARD.

Azo dyes. CHEM. Fabr. ROHNER A.-G. PRATTELN (Swiss P. 111,495—6, 22.5.24. Addns. to 109,706; B., 1926, 149).—(A) A dye which gives a lake fast to light, and is yellowish-red when produced on the fibre, is obtained by coupling the diazo compound from monobenzoyl-m-phenylenediamine with 2:3-hydroxynaphthoic acid anilide. (B) Coupling the diazo compound from p-benzamido-o-toluidine with 2:3-hydroxynaphthoic acid anilide gives a fast, red lake (bluish-red when produced on the fibre). A. COULTHARD.

Preparation of hydroxy-compounds [dyes] of the dibenzanthrone series. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING, Assees. of K. SCHIRMACHER and K. ZAHN (G.P. 414,924, 6.2.23. Addn. to 414,203; B., 1925, 799).—Benzanthrones having hydroxy-groups in the 3-, 6-, or 7-position to the CO-group of the naphthalene nucleus, are fused with alkali. The hydroxybenzanthrone obtained by heating 3-methoxy-1-benzoylnaphthalene with aluminium chloride gives a blue vat cotton dye. The products can also be used as intermediates. A. COULTHARD.

Production of dyestuff emulsions. C. E. J. GOEDECKE, and COLLOISIL COLOUR CO., LTD. (E.P. 245,678, 9.4.25; cf. E.P. 241,331, B., 1925, 983).—Dyes such as Brilliant Green or Malachite Green, in solution, are converted into colloidal emulsions or suspensions by working up with non-lake-forming materials, such as oil, fat, water-glass, soap, dextrin, starch, or glue in a colloid mill. In this way the dyestuff acquires an increased staining power beyond what it possesses when thickened in the usual way. For example, 1 lb. of Brilliant Green base is converted into chloride with the exact amount of hydrochloric acid required, and the solution made up to 2 lb. with water, 2 lb. of mineral oil are added, and the mixture is emulsified in a colloid mill. The products are used in calico printing and in the preparation of pigments. A. COULTHARD.

Sulphonating β-naphthol. M. L. CROSSLEY and G. S. SIMPSON (U.S.P. 1,570,046, 19.1.26. Appl., 14.10.22).—β-Naphthol is sulphonated with sulphuric acid in the presence of an oxy-compound of boron. H. ROYAL-DAWSON.

New dyestuffs of the pyrone series. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER U. CO. (E.P. 247,003, 31.1.25).—See U.S.P. 1,532,790; B., 1925, 494.

Sulphurising organic compounds [for production of sulphur dyestuffs]. F. W. ATACK (U.S.P. 1,571,181, 2.2.26. Appl., 22.1.23).—See E.P. 196,993; B., 1923, 647 A.

Solid stable diazo compounds. BADISCHE ANILIN- U. SODA-FABR., Assees. of F. GÜNTHER and F. LANGE (U.S.P. 1,572,715, 9.2.26. Appl., 22.7.24).—See E.P. 238,676; B., 1925, 840.

Dyes and dyeing (E.P. 245,587).—See VI.

Solutions of dyes (G.P. 419,223).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Bacterial decay of textile fibres. II. Deterioration of artificial silk through the action of micro-organisms. A. C. THAYSEN and H. J. BUNKER (Biochem. J., 1925, **19**, 1088—1094).—Four samples of artificial silk were tested for their resistance to the action of cellulose-decomposing micro-organisms. They were submitted to three tests: (1) the samples were placed anaerobically in a nutrient solution favouring the development of cellulose-decomposing bacteria; (2) the samples were buried on frames vertically in a light garden soil reaching to a depth of 30 cm., and (3) the samples were submerged on fixed frames in sea-water. Cellulose acetate silk was found to be the most resistant of the four samples tested. The varying degree of resistance among the other three types were in the following order: nitro-silk, viscose silk, and cuprammonium silk. The reasons for the different rates of destruction are discussed. S. S. ZILVA.

See also A., Mar., 241, **Nature of solutions of cellulose in cuprammonium hydroxide** (NEALE). 243, **Swelling and dispersion of colloidal substances in ether–alcohol mixtures** (MARDLES).

Aeroplane dopes. DESCHIENS.—See XIII.

PATENTS.

Apparatus for the manufacture of artificial threads. N. B. GRILLET, Assr. to SOC. POUR LA

FABR. DE LA SOIE "RHODIASETA" (U.S.P. 1,571,474, 2.2.26. Appl., 5.3.24).—See E.P. 233,384; B., 1925, 587.

Apparatus for drying textile or fibrous material. H. HAAS (E.P. 246,655, 17.1.25).

Emulsifying or mixing apparatus [for preparing size for paper]. R. B. BEST (E.P. 246,608, 17.11.24).

Coating welding electrodes (E.P. 230,831).—See X.

Solutions of cellulose esters (G.P. 419,223).—See XIII.

Tans from sulphite-cellulose (U.S.P. 1,563,010).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyes and dyeing. [Preparation of derivatives of leuco-compounds of vat dyestuffs.] J. I. M. JONES, B. WYLAM, J. MORTON, and MORTON SUNDOUR FABRICS, LTD. (E.P. 245,587, 12.7 and 30.7.24).—*Derivatives* of leuco-compounds of vat dyestuffs, particularly those of the anthraquinone series, *e.g.*, leucoflavanthrone, leucoindanthrone, and leucoanthraquinone-2 : 1-naphthacridone (Caledon Red BN), for use in dyeing and printing, are prepared by treating the leuco-compounds with alkyl chlorosulphonates, *e.g.*, methyl or ethyl chlorosulphonate. For example, 13 pts. of methyl chlorosulphonate are added during ¾ hr. to a well stirred suspension of 5 pts. of dry leucoflavanthrone in a mixture of 25 pts. of carbon disulphide and 8 pts. of pyridine at 20°. When addition is complete, the temperature is raised to 80° for 10 min., and the product is precipitated by shaking with water, and removed. The product dissolves in dilute alkalis, yielding a blue-violet solution which gives bright blue shades on cotton without the use of a vat. The dry leuco-compounds are prepared by precipitation with acids from alkaline vats, the precipitated material being washed with air-free water and alcohol, and dried in a steam oven, or over sulphuric acid *in vacuo*, or in a rarefied atmosphere of a non-reacting gas, preferably below 50°.
L. A. COLES.

Stable vat dye printing pastes. FARBENFABR. VORM. F. BAYER U. CO., Assees. of R. FISCHER (G.P. 418,990, 7.5.24).—The pastes contain zinc hyposulphite-aldehyde compounds and alkali. Prints obtained with these pastes are stable in the air and can be kept for weeks before steaming.
A. COULTHARD.

Production of fast shades on wool with indigo or indigo derivatives. DURAND U. HUGUENIN A.-G. (G.P. 419,061, 25.12.23).—Wool is allowed to take up dehydroindigo-bisulphite compounds from an acid bath, after which the compounds are converted into the corresponding indigoid parent substances by known methods. For the development of the colour, the wool, dyed with the bisulphite compounds, is treated with dilute sulphuric acid, or with ammonia or alkali carbonates at ordinary or at higher temperatures.
A. COULTHARD.

Dyeing cellulose acetates. R. CLAVEL (U.S.P. 1,571,320, 2.2.26. Appl., 5.10.22).—See E.P. 199,754; B., 1923, 826 A.

Azo dyes (Swiss P. 111,123 and 111,497–9).—See IV.

Azo dyes (Swiss P. 111,495–6).—See IV.

Dyestuff emulsions (E.P. 245,678).—See IV.

Dyeing wood (U.S.P. 1,570,575).—See IX.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Unsoundness of a large chlorine tank at the Wilhelmsburg tin works. F. BÖRNER (Chem.-Ztg., 1926, 59, 85—86).—A large tank containing 10—12 tons of liquid chlorine was allowed to stand idle in the Wilhelmsburg tin works for several years. As a result serious corrosion of the valve on the manhole cover took place and it became necessary to empty the tank without delay. In order to do this a drill working in a gas-tight tube connected to a horizontal valve was made and fixed on the top of the tank, using lead washers to make the joint gas-tight. By operating a lever the tank was drilled and the chlorine delivered through the valve into milk of lime. The time consumed in this operation was so great that the valve corroded and a serious leakage took place. This was finally stopped after much trouble by reducing the pressure in the valve by pushing the drill further into the tank and affixing new washers. This hole was then abandoned and a second valve inserted in the same way, except that the drill fitted into a conical chamber so that in case of further accidental leakage the hole could be stopped efficiently by pushing in the drill.
A. R. POWELL.

See also A., Mar., 217, **Critical potentials of hydrogen in presence of catalytic nickel and copper** (WOLFENDEN). 232, **Heat capacities of metal oxides** (PARKS and KELLEY); **Melting point of graphite** (RYSCHKEWITSCH and MERCK). 235, **Partial pressures of water vapour and sulphuric acid vapour over concentrated sulphuric acid at high temperatures** (THOMAS and BARKER). 236, **Solubilities of sodium, potassium, and calcium ferrocyanides** (FARROW). 237, **Barium dithionate, and solubility of calcium dithionate** (ISHIKAWA and KIMURA). 240, **Stability of carbon dioxide solutions** (KLING and LASSIEUR). 244, **Mass action equation for condensed gases, with application to Haber equilibrium data** (GILLESPIE). 246, **Basic salts of copper** (BRITTON); **Dissociation and specific heats of chlorine and hydrogen chloride at high temperatures** (WOHL and KADOW); **Thermal dissociation of sodium carbide** (GUERNSEY and SHERMAN). 249, **Action of boric acid on alkali carbonates in solution**

(VANZETTI). 250, **Union of carbon monoxide and oxygen in contact with nickel, copper, and their oxides** (BONE and ANDREW). 256, **Double salt of ammonium nitrate and sulphate** (WÖHLER and SCHÄFFER). 257, **Thiosulphuric acid** (RIESENFELD and GRÜNTHAL). 258, **Hypochlorous acid and alkali hypochlorites** (MÜLLER). 260, **Purification of phosphoric oxide** (FINCH and FRASER) ; **Determination of small amounts of bromine** (HIBBARD).

PATENTS.

Concentration of nitric acid. M. KALTENBACH (F.P. 594,865, 7.3.25).—Concentrated sulphuric acid is introduced at the top of a column and meets halfway down a dilute solution of nitric acid. The heat evolved volatilises most of the nitric acid, which is carried upwards and condensed. The mixture passes to a heated cascade where the remaining nitric acid is volatilised, and the sulphuric acid is concentrated ready for re-introduction at the top of the column.

R. B. CLARKE.

Preparation of sulphuric acid of high concentration. METALLBANK U. METALLURGISCHE GES. (G.P. 419,559, 27.2.24).—Sulphuric acid of 66° B. (*d* 1·84) and oleum are prepared by combining the contact process with the processes described in G.P. 370,369, 370,853, and 378,610 (cf. E.P. 149,648 and 184,966 ; B., 1921, 693 A ; 1922, 858 A).

S. S. WOOLF.

Purifying phosphoric acid and preparing pure phosphates. A.-G. FÜR CHEM. PROD. VORM. H. SCHEIDEMANDEL, Assees. of H. J. BRAUN (G.P. 420,173, 21.5.22).—Impurities are precipitated from crude phosphoric acid by addition of sufficient ammonia and then, according to whether phosphoric acid or ammonium phosphate is desired, the ammonia is wholly or partly removed by heating under normal or reduced pressure. The metaphosphoric acid or metaphosphate formed is converted into the ortho-compound by the usual method. A. COUSEN.

Treatment of alkali liquors [obtained in purification of lead]. H. HARRIS (E.P. 245,479, 8.8. and 20.9.24).—Sodium stannate is almost insoluble in cold sodium hydroxide solution (508 g. per litre) or in sodium chloride solution (400 g. per litre). Sodium arsenate is soluble in these solutions but becomes insoluble in a solution containing 280 g. of sodium hydroxide and 124 g. of sodium chloride per litre whereas sodium stannate is soluble. Sodium antimonate is insoluble in hot sodium hydroxide solution of 508 g. per litre concentration. Methods based on these facts, and apparatus, for separating the impurities sodium stannate, sodium arsenate and, when present, sodium antimonate, from spent caustic soda used for the purification of lead (cf. E.P. 189,013 ; B, 1923, 60 A) are outlined. L. M. CLARK.

Carrying out circuit reactions [ammonia synthesis] under very high pressures. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 245,553, 12.11.24).—The difficulty of providing effective packing for circulating pumps moving gases at pressures of 100 atm. and above is avoided by placing the pump, of multistage centrifugal type, and motor, within the high-pressure circuit. In the case of ammonia synthesis a partition, through which the driving shaft passes, separates the pump and motor. This allows only the fresh gas mixture, containing no ammonia, to come in contact with the copper parts of the latter.

C. IRWIN.

Production of catalysts for the synthesis of ammonia. I. W. CEDERBERG, Assr. to NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (U.S.P. 1,570,333, 19.1.26. Appl., 14.7.24).—Compounds of metals of the iron group are mixed with cyanides in liquid, anhydrous ammonia and heated in a non-oxidising atmosphere, after removing excess of ammonia. H. ROYAL-DAWSON.

Hydrogen–nitrogen mixture for ammonia synthesis. CHEM. FABR. KALK G.m.b.H. (G.P. 419,860, 12.11.22).—The gaseous mixture is prepared from a mixture of water-gas and a gas containing nitrogen by burning the carbon monoxide contained therein by steam. Gas obtained by gasifying peat, lignite or similar fuels, is used as the nitrogen-containing gas, without previous condensation of steam. A. COUSEN.

Production of ammonia. E. DUGOUJON (F.P. 594,712, 3.6.24).—A mixture of 1 pt. of air and 2·5 pts. of steam is passed over glowing coal, which is impregnated with a mixture of salts, *e.g.*, chloride, sulphate, carbonate of sodium, magnesium, calcium, etc., obtained by calcining vegetable substances, such as seaweed. The water-gas produced contains 5% of ammonia, which is absorbed in sulphuric acid. The salts present in the coal act as catalysts.

R. B. CLARKE.

Working-up of crude ammoniacal liquor. F. RASCHIG (G.P. 420,498, 11.6.24).—In a continuous process for obtaining pure ammonia solution, the crude ammoniacal liqour is preheated by the spent ammonia-free liquor and then passed down a column which is packed so as to present a large surface area, and is heated to 96°. Volatile acids are thus removed. The liquor is then passed down another column containing similar packing. Steam is injected from the bottom and the ammonia is carried upwards and condensed in a cooling apparatus. The small quantity of fixed ammonia is lost but complications due to addition of lime are avoided and ammonia solution of about 20% concentration is obtained directly. R. B. CLARKE.

Recovery of thiocyanates and thiosulphates. M. DARRIN, Assr. to KOPPERS CO. (U.S.P. 1,570,047, 19.1.26. Appl., 16.1.23. Renewed 11.6.25).—A solution containing sodium thiocyanate, sodium thiosulphate and finely divided impurities, is concentrated till its boiling temperature is 130—135°/760 mm. and is then cooled to a temperature between the transition points of sodium thiosulphate pentahydrate and sodium thiocyanate hydrate. On

seeding with crystals of hydrated sodium thiosulphate a gelatinous precipitate of sodium thiosulphate is formed which entraps the impurities; the solution is filtered and the filtrate concentrated till its boiling temperature is near that of a saturated solution of sodium thiocyanate, and cooled to cause crystallisation of anhydrous sodium thiocyanate, which is then separated from the mother liquor. H. ROYAL-DAWSON.

Manufacture of sodium thiosulphate. H. HOWARD, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,570,253, 19.1.26. Appl., 13.1.25).—In the production of sodium thiosulphate by the interaction of sodium sulphite with sulphur, the sulphur is used in gaseous form. H. ROYAL-DAWSON.

Recovering vanadium from petroleum hydrocarbons. A. OBERLE (U.S.P. 1,570,170, 19.1.26. Appl., 25.6.24).—The volatile constituents of the hydrocarbons are driven off and the residue is leached with a solvent to recover vanadium compounds. L. M. CLARK.

Ammonia-soda process. H. A. GALT, Assr. to PITTSBURGH PLATE GLASS Co. (U.S.P. 1,570,299, 19.1.26. Appl., 1.5.22).—Crude ammonia, concentrated brine, and carbon dioxide gas of low concentration are brought into contact with each other to form ammonium carbonate without material precipitation of sodium bicarbonate; carbon dioxide gas of high concentration is then passed into the mixture to precipitate sodium bicarbonate, which is subsequently calcined to yield soda ash and carbon dioxide gas of high concentration. H. ROYAL-DAWSON.

Sodium carbonate and ammonium chloride. E. A. E. WACHÉ (F.P. 594,695, 31.5.24).—The reaction between ammonium carbonate and sodium chloride is carried out in a cold saturated solution of $(NH_4)_2SO_4,Na_2SO_4,4H_2O$, with a solution containing a fixed concentration of ammonium carbonate. A. COUSEN.

Ammonium chloride and sodium sulphate. E. A. E. WACHÉ (F.P. 594,696, 31.5.24).—Calcium sulphate and ammonium carbonate are added at 65—70° to a solution of $(NH_4)_2SO_4,Na_2SO_4,4H_2O$, saturated in the cold. The precipitated calcium carbonate is removed, sodium sulphate is added, and by sudden cooling the double sulphate is crystallised out, this being converted into ammonium chloride and sodium sulphate by means of a solution of sodium chloride, saturated at 30—35°. A. COUSEN.

Separation of didymium from cerium. DEUTSCHE GASGLÜHLICHT-AUER-GES. (F.P. 594,783, 15.1.25. Conv., 18.1.24).—Cerium peroxide is produced electrolytically in solutions of cerium salts. A. COUSEN.

Potassium salts from sea-water. E. NICCOLI (F.P. 594,904, 10.3.25).—Sea water which has been caused to deposit most of its sodium chloride by evaporation in the sun to 29° B. (*d* 1·25) is further concentrated to 34° B. (*d* 1·31) in open pans by solar heat, when a deposition of unimportant salts is effected. The decanted solution is transferred to another pan and evaporated to 37—38° B. (*d* 1·345—1·36). A mixture of potassium, sodium, and magnesium salts is deposited which is removed and dried. This is dissolved in cold water and mixed with a saturated solution of magnesium sulphate of 33° B. (*d* 1·3). On exposing to solar heat $K_2SO_4,MgSO_4,6H_2O$ containing 1·0—1·5% of sodium chloride is deposited. The addition of magnesium sulphate may be avoided by suitable evaporation. A solution of the double salt is treated with the calculated quantity of milk of lime and filtered. Magnesium hydroxide and calcium sulphate are removed, and the potassium sulphate in the filtrate is crystallised out by solar heat. It has a purity of 97—98%. R. B. CLARKE.

Stable alkali polysulphide solutions. E. GLÜCKSMANN (G.P. 419,910, 24.9.24).—To the solution are added as stabilisers substances or mixtures of substances containing one or more OH-groups, substances capable of increasing the viscosity of the solution, protective colloids, or substances capable of forming complex compounds with certain gases, such as carbon dioxide or hydrogen sulphide. A. COUSEN.

Stable solution of unstable halogen compounds of heavy metals. H. TRUTTWIN (G.P. 420,391, 31.5.24. Conv., 26.5.24).—Stable solutions of unstable halogen compounds of heavy metals, particularly of uranium and vanadium, or of corresponding complex compounds, are obtained by double decomposition in aqueous or alcoholic solution. The compounds, UO_2I_2 and UO_2BiI_5, are mentioned as examples. A. COUSEN.

Lead carbonate. BADISCHE ANILIN U. SODA-FABR., Assees. of L. BUB (G.P. 420,638, 30.12.23. Addn. to 336,767; cf. B., 1921, 583 A).—Lead carbonate is prepared by treating lead sulphate with ammonia and carbon dioxide, using an excess of the former over the latter gas. A. COUSEN.

Preparation of hydrogen sulphide. C. EHRENBERG, H. WIEDERHOLD, C. KRUG, M. G. HOLSBOER, K. FISCHER, and STUDIENGES. FÜR AUSBAU DER IND. (G.P. 421,267, 11.11.23).—Sulphides or hydrosulphides are decomposed by humic acid or substances containing humic acid, *e.g.*, peat, with or without application of heat and pressure. Sodium chloride etc. may be added to the reaction mixture. S. S. WOOLF.

Preparation of carbides. C. EHRENBERG, H. WIEDERHOLD, C. KRUG, M. G. HOLSBOER, K. FISCHER, and STUDIENGES. FÜR AUSBAU DER INDUSTRIE (G.P. 421,268, 30.11.23).—Humic coal, or humus-containing substances, such as peat, are mixed with lime and a suitable amount of water. The mixture, which quickly sets, is coked, yielding gas, tar, and ammonia as by-products, and the residue, which contains carbon and lime intimately mixed, is

utilised as raw material for production of calcium carbide or calcium cyanamide. R. B. CLARKE.

Preparation of table salt from powdered rock salt. GEWERKSCHAFT EINIGKEIT I. (G.P. 421,788, 13.9.24).—The powdered rock salt is suspended in a saturated salt solution, or is washed with water and then freed from the greater part of the washing liquid, a film of solution being thus formed around each of the grains. The resulting mass is quickly dried and a compact conglomerate is obtained, which is again disintegrated by a milling process. R. B. CLARKE.

Extraction of helium from gases. R. R. BOTTOMS (U.S.P. 1,569,943, 19.1.26. Appl., 27.12.24). —The compressed gaseous mixture containing helium is led through one series of passages in a countercurrent heat exchanger, and then into a condenser where the constituents, including the greater part of the helium, are liquefied at high pressure by cooling to the lowest temperature reached in the process. On reducing the pressure by passing the resulting liquid through a throttle valve into a collector, the dissolved helium is released and passing into the gaseous phase is withdrawn. The liquids then pass into a condenser where they are again evaporated to the gaseous state, the vapour passing through a second series of passages in the heat interchanger at low pressure. H. ROYAL-DAWSON.

Decolorising diamonds. L. WOLFRUM (G.P. 419,476, 27.7.21).—Coloured diamonds are embedded in powdered carbon (preferably wood charcoal) and heated, first with exclusion of air, and then to redness in a stream of hydrogen. The smallest adhering carbon particles are then removed by treating with nitric acid and potassium chlorate, warming, and washing with water. A. COUSEN.

Obtaining pure sulphur. BADISCHE ANILIN- U. SODA-FABR., Assees. of F. WINKLER (G.P. 420,238, 2.10.24; Addn. to 358,700).—Pure sulphur is prepared as described in the chief patent (cf. E.P. 177,103; B., 1922, 373 A) from impure ammonium polysulphide solution. By varying the conditions a coarser form of sulphur may be obtained. In place of, or together with, the protective colloid, a surface-active material is used. A. COUSEN.

Purification of sulphur. BADISCHE ANILIN- U. SODA-FABR., Assees. of L. FRIEDERICI (G.P. 421,785, 22.7.24).—Sulphur is melted with water under pressure, in presence of dilute solutions of alkalis or alkaline compounds. S. S. WOOLF.

Obtaining hydrochloric acid free from arsenic. E. SCHMIDT, Assr. to GES. FÜR CHEM. PRODUKTION (U.S.P. 1,571,648, 2.2.26. Appl., 2.5.24).—See E.P. 216,129; B., 1925, 70.

Purification of crude ammoniacal liquors. A. WEINDEL, Assr. to ZECHE M. STINNES (U.S.P. 1,571,986, 9.2.26. Appl., 23.7.24).—See E.P. 230,705; B., 1925, 356.

Removing hydrogen sulphide from gas (E.P. 245,575).—See II.

Recovering aluminium chloride from oil residues. (U.S.P. 1,568,171).—See II.

Lead oxide from storage-battery plates. (U.S.P. 1,570,438).—See XI.

VIII.—GLASS; CERAMICS.

Influence of boric oxide on the properties of chemical and heat-resisting glasses. I. W. E. S. TURNER and F. WINKS (J. Soc. Glass Tech., 1925, 9, 389—405).—Boric oxide was introduced into a typical Kavalier's glass (soda-potash-lime silicate) to replace the silica weight for weight and in successively increasing amounts up to 40% B_2O_3. The general formula of the glasses obtained was SiO_2 (75·82—x)%, B_2O_3 x%, CaO 8·56%, Na_2O 6·86%, K_2O 7·9%, with small amounts of Al_2O_3, Fe_2O_3, and MnO. The average loss of B_2O_3 in the melting operation was 15·8%. Substitution of B_2O_3 for SiO_2 caused an increase of the rate of melting at 1400°, but at temperatures below 1350° segregation occurred, giving, for a time, an upper layer of B_2O_3. With substitution of B_2O_3 for SiO_2 there was increase of pot attack, and the fluidity of the glass and the rate of setting also increased. Continued replacement of the SiO_2 by B_2O_3 caused a rise in annealing temperature until a maximum was attained at 14·5% B_2O_3, followed by a rapid decrease. Similarly, the density gave a maximum at 13·8% B_2O_3, and the refractive index at 26% B_2O_3. The dispersion $n_F - n_C$ remained practically constant over the whole range, and the ν value (61—65) slowly increased with rise of B_2O_3 content. A. COUSEN.

Optical glass. H. HEINRICHS and W. TEPOHL (Glastech. Ber., 1925, 3, 213—222; Chem. Zentr., 1926, I., 761—762).—Barium flint, flint, barium crown, and a series of other glasses are liable to attack by acids with formation of white iridescent spots. A measurement of durability to acids is obtained by heating 1 c.c. of the glass powdered to 0·15—0·3 mm. mesh with 20 c.c. of 0·5% acetic acid. The dissolved barium or lead is precipitated by boiling with 10 c.c. of 0·5N-potassium dichromate and after 1—2 hrs. is collected on a tared paper and weighed. The chromic acid in the filtrate is determined iodometrically. Tendency towards spotting increases rapidly with increase of lead oxide and barium oxide. Glasses containing boric anhydride with little alkali are liable to attack. In baryta crowns which contain lead oxide, barium oxide, and sometimes small amounts of boric anhydride the tendency to spotting is small, and from non-spotting crown glass no salts can be dissolved. Glasses containing antimony give an acid-soluble portion which is analytically determined. A classification of optical glasses into five groups based on the results of the test is given. The test gives no indication of susceptibility towards attack by salt solutions, hydrogen sulphide, etc. A. COUSEN.

Factors involved in the preheating of glass pots, with special reference to moisture control. W. W. OAKLEY (J. Amer. Ceram. Soc., 1926, 9, 23—28).—Mechanical strains are induced in the material of glass pots owing to varying conditions of humidity—and therefore rate of drying—outside and inside the pots. To minimise the effect of this, the atmosphere in the pot arch during the preheating process must be kept as humid as possible by the injection of steam etc. Natural gas is a very suitable fuel, since it gives the wettest flue gas. F. SALT.

Sillimanite in glass furnace practice. F. G. CLARK and W. J. REES (J. Soc. Glass Tech., 1925, 9, 383—388).—The linear shrinkage after burning at 1500°, the refractoriness, and the refractoriness under a load of 50 lb. per sq. in., of mixtures of sillimanite with dextrin, shellac, colloidal aluminium silicate, and ball clay were determined. Examination of the workability of the mixtures indicates that it would be best to use not less than 20% of ball clay as a bond for pot-making purposes, and 25% was finally adopted. The mixture was best when soaked for not less than four weeks before making up. From such a mixture a covered pot, 54×39×43 in., was successfully made in 16 days, and it was quite dry in 6 weeks. The total drying contraction was 1 in 100. Normal methods of arching and setting of the pot were used, but it only survived one week of melting operations, that is, two founds. Failure was due to a crack in the bottom, probably due to shock received in transferring from pot room to arch. Glass corrosion on both the pot and a ring of the same material was negligible. The cost of the pot was approximately 3 times that of a similar clay pot. Bricks made from sillimanite–bauxite–ball clay mixtures after firing at 1500° had a shrinkage of 2·8% and had a refractoriness equal to that of sillimanite itself. The high resistance of sillimanite–ball clay bricks to corrosion by glass was proved by immersing these for a period of 6 weeks in a glass pot. A. COUSEN.

Sillimanite bricks and kaolin-sillimanite mixtures. H. S. HOULDSWORTH (J. Soc. Glass Tech., 1925, 9, 316—321).—Addition of sillimanite, of a grading as supplied commercially, to kaolin and to fireclay reduces the shrinkages on drying and on firing (to cone 14), whilst it increases the porosity. The refractoriness of the fireclay mixtures is also increased. Kaolin–sillimanite mixtures containing less than 60% of sillimanite are not so resistant to the solvent action of soda-lime glass as kaolin itself, whilst these mixtures are apparently more resistant than corresponding fireclay–sillimanite mixtures, the temperature at which vigorous interaction takes place being appreciably higher with the first-named mixtures. Kaolin–sillimanite mixtures give a regular reversible thermal expansion not affected by variations of the amount of sillimanite present. A commercial sillimanite brick was found to possess a porosity of 31·2% and a fusion point higher than that of cone 37. Ground samples of the brick heated in a gas-fired furnace for 2 hrs. at 1410° and then for 2 hrs. at 1500° gave a mean contraction at the lower temperature of 0·12% and at the higher a further 0·13%. The reversible thermal expansion from 15° to 1000° was about 0·45%. The brick possessed high refractoriness and resisted attack by soda-lime glass to a considerable degree. A. COUSEN.

Commercial sillimanite as a refractory material. I. Choice of bonding material and of grain size. A. COUSEN and W. E. S. TURNER (J. Soc. Glass Tech., 1925, 9, 334—346).—Microscopical examination of a sample of commercial sillimanite indicated much change of structure from the raw unfired material, suggesting the formation of a considerable proportion of mullite. Chemical analysis showed the presence of more than 6% of corundum, and the alumina content of the rest lay between that required for sillimanite and that for mullite. Comparative tests of plasticity, mechanical strength, and friability and also of porosity of mixtures of three different grades of the commercial sillimanite with ball clay, Stourbridge clay, and bentonite as bonding agents were carried out. Ball clay was the best bonding agent, the bentonite mixtures, although more plastic, giving a much greater drying shrinkage and a greater porosity. Comparison of ball clay mixtures amongst themselves showed that the one with 30 pts. of clay to 100 pts. of fine sillimanite (79% through 120-mesh) gave the least porous material of the mixtures tried. A mixture with 4 pts. of fine and 2 pts. of the coarser fractions gave a porosity only 2% greater than that of the fine material, was little less plastic, and gave equally good results in melting operations. A. COUSEN.

Commercial sillimanite as a refractory material. II. Porosity, density, and mechanical strength of sillimanite–ball clay mixtures. A. COUSEN and W. E. S. TURNER (J. Soc. Glass Tech., 1925, 9, 347—356).—Two series of sillimanite mixtures, the one with the bulk of particles lying between 50- and 70-mesh, the other with all particles below 120-mesh, and both with ball clay as a bond, were compared as to porosity, density, and mechanical strength. The proportion of bond used was varied in each case to give 15, 30, and 40 pts. per 100 pts. of sillimanite. The tests served to show the advantage of the finer grade material, since this gave a lower porosity on firing and an increased mechanical strength. In all cases shrinkage and mechanical strength increased continuously with rise of firing temperature from 100° to 1400°, whilst the porosity decreased. In the fine sillimanite mixtures two zones of change were noted, namely 800—1000° and above 1300°, the former coinciding with that of the rapid shrinkage of ball clay, the latter probably indicating conversion of the mass into mullite. A mixture containing at least 30 and probably 40 pts. of ball clay per 100 pts. of sillimanite, and having much of the latter in a fine-grained form, proved most satisfactory, from the point of view of the properties examined, for the preparation of refractory articles. A. COUSEN.

Commercial sillimanite as a refractory material. III. Resistance to corrosion by glass. A. COUSEN, S. ENGLISH, and W. E. S. TURNER (J. Soc. Glass Tech., 1925, 9, 357—370).—Melting tests of soda-lime and potash-lead glasses in small sillimanite–ball clay and sillimanite–Stourbridge clay pots of compositions as studied in previous papers (cf. preceding abstracts), showed that the glass dissolved less iron oxide from the pots than it did from clay pots. The tests led to the choice of the mixture: fine sillimanite (through 100-mesh sieve) 5 pts., medium sillimanite (70- to 100-mesh) 1, coarse sillimanite (30- to 70-mesh) 1, ball clay (finely ground) 3 pts., for making larger pots for further work. These larger pots dried much more rapidly than did clay pots of the same size, the drying shrinkage being small, namely 1 in 39, and the firing shrinkage was extremely small. In the furnace, the pots were successfully used for relatively long periods at temperatures of 1400—1500° and with severe temperature drops at week ends, without signs of sagging or losing shape. Glass worked from the pots was of very varying types and composition, but there was a distinct absence of stones. Analysis of the glasses proved them to have abstracted, on the average, distinctly less iron oxide from the pots than would have been dissolved from clay pots. The alumina content of the glasses, however, was fairly large and indicated distinct general corrosion, although this corrosion was more uniform and less marked than with similar fireclay pots. A. COUSEN.

Mineralogy of clay. I. J. S. MCDOWELL (J. Amer. Ceram. Soc., 1926, 9, 55—60).—In a review of the literature on the microscopical examination of clays, the constituents of clays are grouped and discussed under three headings, viz., the essential, the major accessory, and the minor accessory ingredients. F. SALT.

Rational analysis of clay. II. J. S. MCDOWELL (J. Amer. Ceram. Soc., 1926, 9, 61—65).—A review of the literature. The methods of rational analysis are based on the inaccurate assumption that clays consist mainly of kaolinite, quartz, and undecomposed felspar, whereas microscopical examination shows that many clays contain hydromica, and not felspar. Greater accuracy in the calculation of rational analyses might be possible by the use of data supplied by time–temperature curves and microscopical investigation. F. SALT.

An eighteen months' high-temperature test on refractory test specimens. F. H. RIDDLE and A. B. PECK (J. Amer. Ceram. Soc., 1926, 9, 1—22).—The effect was studied of time, temperature, kiln atmosphere, etc. upon a number of standard bodies and experimental mixtures, which had been allowed to remain in various parts of a Dressler muffle kiln for a period of eighteen months. The kiln was fired to cone 17—18, and the atmosphere was mainly of an oxidising nature. The construction of the kiln is described. At about 600°, silicon carbide specimens expanded to nearly twice their original size, much cristobalite being formed. At the highest temperature (above 1446°) swelling was not so apparent, but volatilisation was very pronounced and oxidation to cristobalite was almost complete. Mullite was found in varying amounts in most of the specimens. Porcelain sparking-plug bodies exhibited no change in microstructure up to 1200°, but at 1296° the mullite crystals had increased somewhat in size. At 1462° their average size was about four to five times larger than the original. No corundum was developed. At high temperatures the glaze had been absorbed or volatilised. Sections of pyrometer tubes were affected in a similar manner up to 1050°, but at the highest temperature crystal development was more marked. Pyrometer tubes were less refractory, and showed a tendency to blister and craze. The microstructure of silica bricks taken from various parts of the hot zone indicated definite variations in temperature, both vertically and horizontally, within that zone. Silicious bonding material proved more suitable than fireclay or chromite. A silicon carbide mortar was oxidised to cristobalite in the hot zone, the resulting expansion giving a very tight joint. The incidental formation of three artificial minerals was observed. Volatilised silica from the silicon carbide and silica bricks in the hot zone was deposited in the water-smoking zone as silica glass. Crystals composed of a mixture of Fe_3O_4 and Fe_2O_3 were found in the cooling zone under conditions indicating a dissociation temperature of Fe_2O_3 lower than hitherto accepted. Lithophysæ composed of cristobalite were formed, by oxidation, on many silicon carbide specimens in the hot zone. F. SALT.

Deformation study of various aluminosilicates and borosilicates. K. C. LU (J. Amer. Ceram. Soc., 1926, 9, 29—54).—The literature is briefly reviewed. Two aluminosilicate and three borosilicate eutectics were developed within the limits of 2·5 molecular per cent. differences of the various constituents. The molecular formulæ and approximate deformation temperatures are: PbO: $0{\cdot}254Al_2O_3$:$1{\cdot}91SiO_2$, 650°; ZnO:$0{\cdot}225Al_2O_3$:$0{\cdot}906SiO_2$, 1360°; PbO:$0{\cdot}238B_2O_3$:$0{\cdot}78SiO_2$, 415°; Na_2O:$1{\cdot}29B_2O_3$:$1{\cdot}73SiO_2$, 570°; K_2O:$2{\cdot}11B_2O_3$:$2{\cdot}585SiO_2$, 655°. The relation between these eutectics was studied by plotting their positions, according to molecular percentages, on the triaxial diagram. F. SALT.

Action of gases on porcelain glazes. A. PFAFF (Ber. Deuts. Keram. Ges., 1925, 6, 42—44; Chem. Zentr., 1926, I., 763).—The effects of the individual constituents of a normal mixture of furnace gases upon glazed hard porcelain during burning were investigated. Oxygen gave a rough, ugly effect, which was less pronounced with air and absent with nitrogen. Carbon dioxide caused no faults, but carbon monoxide produced small bubbles, and hydrogen foamy bubbles. A. COUSEN.

Thermal expansion of Jena glass 16^{III}. VAN AGT and ONNES.—See A., Mar., 231.

Patents.

Production of copper mirrors on glass. K. Bamberger and R. Schweizer (G.P. 420,469, 6.2.24).—Copper mirrors may be produced on glass and other non-metallic substances by treating a suspension of cuprous hydroxide in a solution containing 0·5% of free alkali with hydrazine hydrate or sulphate in contact with the glass. The deposits adhere well and have a high lustre. A. R. Powell.

Apparatus for washing clay. K. H. Reichau (E.P. 245,608, 24.1.25).—A conduit formed of tubular members of different diameters increasing stepwise from bottom to top is suspended in a water container. At the top the conduit passes through the floor of a receptacle into which it discharges. The coarse particles separate from the clay suspension as it flows upwards through the conduit and fall through the gaps between the tubular members into the water container. P. B. Robinson.

Preparing clay or body composition for ceramic articles. A. O. Austin, Assr. to Ohio Brass Co. (U.S.P. 1,569,251, 12.1.26. Appl., 1.3.23). —Material is prepared partly as a slip and partly dry, and mixed to provide a body mix having a slightly greater percentage of water than that at which the ceramic articles are formed, the excess water being removed by evaporation. P. B. Robinson.

Manufacturing artificial abrasives. C. J. Brockbank, Assr. to Abrasive Co. (U.S.P. 1,568,873, 5.1.26. Appl., 11.9.20).—Substantially pure crystalline alumina is produced by fusing an aluminous ore with a reducing agent and boric acid. F. Salt.

Method of making crucibles. A. J. Jackman, Assr. to Vesuvius Crucible Co. (U.S.P. 1,570,064, 19.1.26. Appl., 18.3.25).—A crucible for metal-melting operations in which slag is formed has a graphite body adapted to be in contact with the bulk of the metal, whilst the portion which is to be in contact with the slag is made of material which is more resistant than graphite to the action of the slag. W. Clark.

Manufacture of silica bricks. Dr. North A.-G. (G.P. 420,851, 30.10.21).—Powdered quartzite or sand is mixed with the usual binder and with powdered coal, which accelerates the transformation of the quartz into tridymite and cristobalite during firing. A. R. Powell.

Glass. W. C. Taylor, Assr. to Corning Glass Works (U.S.P. 1,572,625, 9.2.26. Appl., 7.1.21).—See E.P. 192,919; B., 1923, 355 a.

Plastic [magnesia compositions]. O. Frosell, Assr. to Williamsport Building Products Co. (U.S.P. 1,572,714, 9.2.26. Appl., 25.3.21).—See E.P. 202,698; B., 1923, 1133 a.

Treating clay (U.S.P. 1,561,971).—See XIV.

IX.—BUILDING MATERIALS.

Patents.

Heat-insulating material. L. G. Wilkening (E.P. 226,522, 8.12.24. Conv., 22.12.23).—Light peat, *e.g.*, peat moss or "fox peat," is coked at 400—600°, producing a fine porous material of low sp. gr. The product may be used alone as an insulating material, or mixed with cementitious or bituminous binding materials to form insulating blocks. B. W. Clarke.

Heat-insulating material. W. T. Irvin, Assr. to Celite Co. (U.S.P. 1,569,755, 12.1.26. Appl., 24.12.24).—Heat-insulating material is prepared by mixing diatomaceous earth and an alkaline-earth, moulding, and then hardening and drying by exposure to large volumes of air under pressure. P. B. Robinson.

Kilns for use in cement manufacture. S. W. Burley (E.P. 245,866, 22.10.24).—Air is forced into the ash-pit of a non-rotary cement kiln and a draught over the slurry bed is induced by an extraction fan situated at the exit end of the kiln. The air supply is controlled so that it may be concentrated at one or more spots on the charge, thus producing more efficient working. The process renders the operation of the kiln independent of atmospheric conditions and increases the output of the kiln as well as enabling low-grade fuel to be burnt. B. W. Clarke.

Cementitious material. N. V. S. Knibbs (E.P. 245,935, 13.1.25).—Lime or magnesia or magnesian lime is subjected to carbonation at a high temperature in order to ensure a degree of carbonation or hydration between definite maximum and minimum limits, which vary with the composition of the raw material. Thus lime containing over 95% of calcium oxide is carbonated to a calcium carbonate content of 30—80%, using waste producer-gas etc. The product when ground possesses good cementitious properties, and may be used either alone or with hydrated lime etc. to form blocks, slabs, etc. B. W. Clarke.

Portland cement. W. Vershofen (G.P. 419,353, 16.1.23).—To the dry, powdered, clay-limestone mixture is added a water-soluble fluoride, capable of increasing the bonding power of the mixture when water is added, while also reducing the firing temperature necessary to produce clinkering in the kiln to about 1000°, owing to the chemical action of the fluoride on the mixture. A. Cousen.

Wood preserving. F. Raschig (E.P. 246,010, 4.6.25).—Sodium silicate (water-glass) is added to a mixture of sodium fluoride and dinitrophenol or dinitrocresol, used as a wood preservative, to prevent the corrosion of iron or steel vessels by the wood-preserving liquid. B. W. Clarke.

Dyeing wood. H. Renner, Assr. to Forest Products Research Corp. (U.S.P. 1,570,575, 19.1.26. Appl., 26.10.23).—The sap in the cellular

tissue of the wood is combined with a mixture of a colouring metal compound and a volatile or unstable chemical which forms unstable salts. The complex salts formed are decomposed when the wood is dried, so that permanent colouring substances are fixed in the cells of the wood. B. W. CLARKE.

Plaster of Paris. E. L. WILSON, Assr. to RUMFORD CHEMICAL WORKS (U.S.P. 1,570,583, 19.1.26. Appl., 22.8.24).—Precipitated gypsum is subjected to pressure to cause coalescence of the crystals, which are converted into a non-compacted extended mass. This is uniformly calcined to produce plaster of Paris. B. W. CLARKE.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Some physical properties of steel and their determination. J. H. ANDREW, M. S. FISHER, and J. M. ROBERTSON (Proc. Roy. Soc., 1926, **A 110**, 391—422).—The electrode potentials and specific resistances of carbon-, chromium-, nickel-, and nickel–chromium-steels, which have been subjected to various types of heat treatment, have been determined as functions of the carbon content of the steel. The variation of specific resistance with time during tempering at 240° has also been investigated for the same steels. The electrode potential (against a solution of ferric chloride as electrolyte) of quenched hypoeutectoid steels is raised, and that of hypereutectoid steels lowered, by raising the quenching temperature. The lowering in the latter case is very marked when the quenching temperature is raised from 1000° to 1100°. The potential of tempered steels rises with the temperature of tempering. The electrode potentials measured are those of α-iron, modified by the presence of carbon, nickel, and chromium in solution; it is lowered, for example, by dissolution of carbon. The specific resistance of hypereutectoid steels is increased, and that of hypoeutectoid steels decreased by raising the quenching temperature. Corrected for the small amounts of silicon, phosphorus, and manganese present, the specific resistance, R, for the carbon steels is given by $R=7{\cdot}5+26{\cdot}7\times$carbon content. In a nickel–chromium steel of given carbon content, the increase in resistance above that of the carbon steel is proportional to the amount of each element present. In the tempering experiments the specimen of steel was immersed in an oil bath at 240°, and its electrical resistance determined at intervals over a period of about 5 hrs. The tempering-resistance curves for all steels quenched at 900°, 1000°, or 1100° were similar, showing an initial rapid decrease in resistance, then a slow fall or an almost constant value. An analysis of these curves permits calculation of the rate at which carbon separates from solution in austenitic and martensitic steels respectively. The usual procedure for investigating the process of tempering, involving periodic cooling of the steel to ordinary temperature to make observations, may lead to erroneous results. During cooling a considerable amount of austenite tempers to martensite; the former can therefore temper in two ways, slowly at a constant temperature, and more rapidly when the temperature is falling. A. B. MANNING.

Determination of cerium in special steels. K. SWOBODA and R. HORNY (Z. anal. Chem., 1926, 67, 386—398).—Slightly low results are usually obtained by precipitation of a slightly acid solution of cerous nitrate with oxalic acid followed by ignition of the cerous oxalate to cerium dioxide. Precipitation as ceric peroxyacetate yields good results even in the presence of chromium and tungsten compounds if the operation is conducted as follows. The solution is treated with 1 g. of tartaric acid, diluted to 200 c.c., and neutralised with ammonia. Two drops of hydrochloric acid, 15 c.c. of 6% hydrogen peroxide, and 10 c.c. of 10% ammonium acetate solution are added and the mixture is boiled; a further 10 c.c. of acetate is added together with a little filter pulp and boiling is continued until the precipitate flocculates well, but care must be taken to avoid the complete destruction of the hydrogen peroxide. The brown precipitate is collected, washed with very dilute ammonium acetate solution, and ignited wet to the dioxide for weighing. In the presence of ammonium salts low results are obtained, in which case precipitation as fluoride is recommended. The procedure is similar to that described above except that precipitation is effected with 20 drops of hydrofluoric acid and a little ammonium fluoride in place of the peroxide and acetate. Ignition of the fluoride, without drying, leaves a residue of the dioxide. The latter method is applicable to the determination of cerium in special steels with the following modified procedure:—2 g. of the steel turnings are dissolved in 60 c.c. of 1 : 1 hydrochloric acid, and nitric acid is added drop by drop to oxidise the iron, avoiding an excess. The hot solution is treated successively with 60 c.c. of 25% tartaric acid solution, 30—35 c.c. of a 10% solution of stannous chloride to reduce the iron, and sodium hydroxide until slightly alkaline. The mixture is cooled and diluted to 500 c.c. in a graduated flask after adding 10 c.c. of alcohol, and filtered rapidly through pleated filters. The filtrate (250 c.c.), which is free from iron and tin, is acidified with hydrochloric acid, heated to boiling, and treated with 2 g. of solid ammonium fluoride. The solution is made just ammoniacal, again acidified with hydrochloric acid, and filtered, after 1 hr., with the addition of filter pulp. The precipitate is washed with a hot 0·3% solution of ammonium fluoride, ignited wet in a platinum crucible, and weighed as CeO_2. A. R. POWELL.

Electroplating on non-rusting iron. J. HAAS and E. R. UNRUH (Metal Ind. N.Y., 1925, **23**, 451—452; Chem. Zentr., 1926, I., 1278—1279).—Before electroplating on non-rusting iron the surface must be pickled in order to remove the thin layer of passive metal so as to obtain a good, strongly adherent deposit. This is best effected by a short electrolytic pickle in concentrated hydrochloric acid

at 60°, or, somewhat less satisfactorily, by heating the article at 60° in 20% sulphuric acid. After this treatment the metal must be plated directly in an acid bath, which prevents the surface from again becoming passive better than an alkaline bath.

A. R. Powell.

Protection of iron by cadmium. H. S. Rawdon (Trans. Amer. Electrochem. Soc., 1926, 49, 21—30. Advance copy).—Cadmium behaves similarly to zinc in preventing the corrosive attack of iron when specimens of iron and steel containing plugs of the protective metals are immersed in $N/2$-sodium chloride solution. The anodic behaviour of cadmium and zinc with respect to iron under the experimental conditions was clearly revealed by the etching action at the surface of the inserted plugs. Whilst the values assigned by most authorities to the electrode potentials of these metals place iron between the other two, the experiments show that for the conditions described, cadmium and zinc stand in the same relation to iron. The rate of etching indicated that the potential difference between zinc and iron when in contact and immersed in sodium chloride solution is considerably greater than that between cadmium and iron under the same conditions. M. Cook.

Nature of the protective film of iron. T Fujihara (Trans. Amer. Electrochem. Soc., 1926, 49, 1—8. Advance copy).—The uncorroded iron outside a drop of water on a polished iron surface is due to a protective film of ferrous hydroxide. The ferrous hydroxide is soluble, forming an alkaline solution, and the film would eventually stop corrosion if carbon dioxide were excluded from the air in which the iron is corroding. The protective film is destroyed by carbon dioxide neutralising the alkaline liquid.

M. Cook.

Determination of silver, gold, and platinum in anode slimes. E. Eckert (Metall u. Erz, 1925, 22, 595—598; Chem. Zentr., 1926, I., 987—988).—Ten g. of the slime are dissolved in nitric acid and, after removal of the insoluble matter, the silver is precipitated with hydrochloric acid and the chloride is weighed. Gold and platinum are determined by scorification and cupellation. The silver bead is dissolved in nitric acid, leaving a residue of gold and most of the platinum. The silver is precipitated as chloride from the solution and the filtrate evaporated to dryness on the water-bath. The residue is dissolved in hydrochloric acid and the solution transferred to a lead capsule, in which it is again evaporated. The capsule and residue are cupelled with the major portion of the platinum and gold and with sufficient silver for the subsequent parting. The bead is parted in sulphuric acid and the residue ignited and weighed. It is then re-alloyed with silver and the bead parted in nitric acid; this is repeated until the gold residue is of constant weight. Platinum is found by difference.

A. R. Powell.

Reverberatory refining of copper. Influence of prolonging the blowing on the impurities in and properties of the metal. W. Heckmann (Metall u. Erz, 1925, 22, 527—546; Chem. Zentr., 1926, I., 1275).—An investigation of the mechanism of the refining of copper scrap in the reverberatory furnace by the addition of black copper, converter copper, and copper scale is described, in the course of which the composition and mechanical properties of samples taken from the bath at ½-hr. intervals have been determined. When the blowing is prolonged so that the metal contains more than 0·8—0·85% O, separation into two layers begins to take place, the upper layer of cuprous oxide then becoming further oxidised and combining with the fluxes on the surface. A. R. Powell.

Influence of cuprous oxide on electrolytic and refined copper. H. Altwicker (Metall u. Erz, 1925, 22, 583—594; Chem. Zentr., 1926, I., 1027). —The mechanical properties of tests taken from charges of various kinds of copper during refining have been examined and correlated with the content of cuprous oxide. Less than 0·23% O has no effect on hot-rolled metal but 0·12% causes corrosion to take place readily in cold-rolled copper. The presence of cuprous oxide has little effect on the tensile strength, but it reduces the ductility very markedly; *e.g.*, the elongation of copper containing 0·01% O is 360% greater than that of copper containing 0·387% O. The hardness of copper increases with the content of cuprous oxide, small proportions of which also improve the Erichsen number. The electrical conductivity decreases with increasing oxygen content, but the pliability is scarcely affected.

A. R. Powell.

Determination of copper, arsenic, and mercury. R. Rosendahl (Chem.-Ztg., 1926, 50, 73—74).—For the determination of arsenic in metals and ores the substance is dissolved in nitric acid and the solution evaporated with sulphuric acid to expel the nitric acid. After dilution an equal volume of hydrochloric acid is added, iron and copper are reduced by addition of stannous chloride to ferrous and cuprous chlorides, and the arsenic is separated by warming the solution at 80—90° with 5 g. of sodium hypophosphite. The washed precipitate is distilled with concentrated hydrochloric acid and a little ferric chloride, the residual solution is diluted to 750 c.c., manganese sulphate is added, and the ferrous chloride formed is titrated with permanganate (0·4475 Fe=As). Alternatively, the arsenic is dissolved in nitric acid and the solution evaporated with sulphuric acid and sulphur to reduce the arsenic acid. After dilution and expulsion of sulphur dioxide the arsenic may be determined as usual with iodine, or, after addition of a known weight of antimonious chloride and 12 c.c. of hydrochloric acid, by titration with permanganate. The addition of antimony ensures a good end-point. Copper may be determined in ores by dissolving in nitric and sulphuric acids a quantity containing not more than 0·2 g. of copper, evaporating to expel the nitric acid, diluting, adding ammonia until alkaline, 6—7 g. of tartaric acid, and 5 g. of sodium hypophosphite. After boiling for 20 min., the precipitated

copper is collected, washed, dissolved in hot acidified ferric chloride solution, and the ferrous chloride formed determined as usual. Mercury is determined by dissolving the metal in nitric acid, evaporating with sulphuric acid, adding 20 c.c. of water, boiling the solution with 50 c.c. of hydrochloric acid to expel arsenic, and finally precipitating the mercury as metal by prolonged boiling with 5 g. of sodium hypophosphite. The precipitate is collected into a globule, dried, and weighed.

A. R. POWELL.

Aluminium-lithium alloys. P. ASSMANN (Z. Metallk., 1926, **18**, 51—54).—Aluminium and lithium form a eutectic with 7·8% Li melting at 598° and a solid solution containing 3·5% Li at 598° and only 2·2% Li at 20°. The Brinell hardness increases with the lithium content to a maximum of 130 with 12·1% Li, but the greatest increase occurs between 2 and 4% Li. On heating to 500°, quenching, and ageing at ordinary temperature all the alloys increase in hardness, especially those containing 0·3% and 2—4% Li; ageing at 200° entirely prevents this increase. The hardness of aluminium alloys containing 0·4—1·0% Li and 4—12% Li is appreciably increased by annealing at 300—400°, and still more so by quenching from 500° and ageing at 18°. Lithium-copper-aluminium alloys increase in hardness by nearly 70% after the latter treatment, the maximum hardness being obtained with 0·4% Li and 4% Cu or with 1·0% Li and 2% Cu. Alloys with 0·4% Li and 2—7% Cu are still further hardened by ageing at 100°, whereas those containing 1·0% Li and 2—4% Cu are slightly softened by this treatment. A. R. POWELL.

Metallic uranium. J. F. GOGGIN, J. J. CRONIN, H. C. FOGG, and C. JAMES (Ind. Eng. Chem., 1926, **18**, 114—116).—Uranosouranic oxide is reduced to finely-divided metal on heating with metallic calcium but the yield is small. A good yield of very pure fused uranium is obtained by reduction of uranium chloride with a 10% excess of metallic calcium; the operation is carried out in an alundum crucible placed inside a nichrome-wound reduction bomb which is placed inside a steel cylinder from which the air is exhausted by means of Hyvac pumps. The product obtained by this method was a hard, brittle metal containing 0·57% Fe, 0·09% C, and 0·03% O when commercial calcium was used, but a much purer product, containing only 0·01% Fe, was obtained by the use of re-sublimed calcium. The ingot showed fine crystalline markings, had a very silvery lustre and convex surface, and appeared to be sound. Uranium chloride for the reduction may be obtained by heating the oxide in a current of chlorine and sulphur chloride to 900—1000°.

A. R. POWELL.

Preparation of antimony-regulus. F. BÖRNER (Metall u. Erz, 1925, **22**, 559—564; Chem. Zentr., 1926, I., 1005).—A mixture of antimony ore and iron is roasted, sodium chloride or carbonate being added to render the slag (ferrous sulphide) liquid. By further treatment, with addition of antimony sulphide, the iron is removed from the crude antimony. The author gives full details of the process and of the analysis of metal and slag.

S. S. WOOLF.

Equilibrium between metals and salts in the molten state; a new form of law of mass action. R. LORENZ (Z. angew. Chem., 1926, **39**, 88—90).—In the melt represented by the equation $M'+M''X \rightleftharpoons M'X+M''$, the metals and salts form two separate phases. The equilibrium state is defined by the relation $[x/(1-x)].[(1-y)/y]=Ke^u$, where x, $1-x$, y, $1-y$, represent the molecular proportions of the reacting and resulting substances, K is a constant, and u is defined by a somewhat complex equation. It is shown experimentally for the system $Pb+CdCl_2 \rightleftharpoons PbCl_2+Cd$ that $\log K$ is an appreciably constant quantity.

W. T. K. BRAUNHOLTZ.

Transformations of certain alloys of aluminium, and influence of deformation. L. GUILLET (Rev. Mét., 1926, **23**, 48—52).—See B., 1925, 995.

Hydrogen-ion control of nickel-plating baths. PARKER and GREER.—See XI.

PATENTS.

Magnetic materials [iron-nickel alloys]. WESTERN ELECTRIC CO., LTD. From WESTERN ELECTRIC CO., INC. (E.P. 245,986, 22.4.25; cf. E.P. 189,410; B., 1923, 101 A).—Iron-nickel alloys containing nickel 80—83%, after suitable heat treatment, have certain electric and magnetic properties which are substantially independent of applied mechanical forces over a considerable range. Under tensions up to 5000 lb. per sq. in., the permeability, the hysteresis loss per cycle, and the resistivity remain practically unaffected. The alloys are particularly suitable as loading material for increasing the inductance of submarine cables. L. M. CLARK.

Annealing sheet steel. G. H. COLE, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. CO. (U.S.P. 1,569,355, 12.1.26. Appl., 21.12.20).—Sheets of magnetic silicon alloy steel are annealed by heating to a temperature not exceeding 700° in an atmosphere containing approximately the same ratio of oxygen and nitrogen as in air. M. COOK.

Alloy [steel]. J. I. MEE (U.S.P. 1,569,996, 19.1.26. Appl., 6.2.25).—An alloy steel containing tungsten, vanadium, chromium, sulphur, copper, and iridium is claimed. A. R. POWELL.

Reduction of iron ores by means of methane or gases containing methane. GELSENKIRCHENER BERGWERKS-A.-G., ABT. SCHALKE (G.P. 421,462, 25.1.24).—In the presence of a small quantity of metallic iron, methane reduces ores containing iron oxide at temperatures below 700°. The metallic iron necessary to start the reduction may be added in powdered form to the ore or, preferably, produced *in situ* by giving the ore a short preliminary treatment with a gas containing hydrogen.

A. R. POWELL.

Cast-iron low in phosphorus. Union de Consommateurs de Produits Metallurgiques et Industriels (F.P. 577,661, 28.4.23; G.P. 421,991, 3.6.23).—Ordinary grey cast-iron is dephosphorised as usual in the converter and the requisite amount of carbonaceous material to regenerate cast iron is added, together with substances such as thermit, ferrosilicon, or ferromanganese which generate heat and raise the temperature of the molten metal so that it can dissolve a greater proportion of carbon. A. R. Powell.

Manufacture of very thin wires. N. V. Philips' Gloeilampenfabrieken (E.P. 235,893, 17.6.25. Conv., 18.6.24).—By using thin wires as the cathode in a glow discharge at such a current density that disintegration occurs, a very thin wire the diameter of which is practically uniform, and may be less than 10 microns, is obtained. The process may be applied to all single-crystal wires. L. M. Clark.

Coating welding electrodes. Siemens-Schuckertwerke G.m.b.H. (E.P. 230,831, 11.3.25. Conv., 12.3.24).—In the preparation of paper-covered electrodes, fluxes or materials for regulating the combustion of the covering may be introduced into the paper or pulp during the course of its manufacture (cf. following abstract). A. R. Powell.

Jacketed welding electrodes. P. C. Rushen. From Siemens-Schuckertwerke G.m.b.H. (E.P. 245,635, 11.3.25).—A jacket or coating for welding electrodes consists of paper, paper pulp, or similar non-conductor which carbonises on heating. The combustion of the jacket during welding operations may be regulated by impregnating it with solutions of substances which aid its combustion (cf. preceding abstract) or by applying such substances either underneath or on the surface of the jacket. A. R. Powell.

Hardening copper. L. Nemitof (U.S.P. 1,569,130, 12.1.26. Appl., 6.2.22).—Copper heated to bright redness is embedded in a mixture containing a silicious substance and calcium phosphate moistened with a hydrocarbon. M. Cook.

Refining copper–nickel matte. R. L. Peek and T. F. Torell, Assrs. to National Trust Co., Ltd. (U.S.P. 1,569,137, 12.1.26. Appl., 26.8.20).—Bessemerised copper–nickel matte is utilised for precipitating copper from copper–nickel sulphate solution by the cementation process. The greater part of the copper is then removed from the partially exhausted matte and the residue is used for the preparation of fresh copper–nickel sulphate solution. L. M. Clarke.

Disintegrating metals in a ball mill or the like. E. J. Hall, Assr. to Metals Disintegrating Co. (U.S.P. 1,569,484, 12.1.26. Appl., 25.3.19. Renewed 3.6.25).—Metal dust is prepared by mechanically disintegrating the metal, while keeping it covered with a lubricating liquid which is inert to the metal, and protects it against the effects of the atmosphere. The lubricating liquid has a b.p. sufficiently high to prevent excessive loss by vaporisation during the disintegration, but not high enough to cause difficulty in its subsequent removal from the metallic powder by evaporation. A. B. Manning.

Alloy for pen-points. M. M. Goldsmith (U.S.P. 1,569,921, 19.1.26. Appl., 16.11.23).—An alloy for tipping the ends of pen nibs comprises 10—20% Ru, 3—10% Ni, and 65—85% Os. A. R. Powell.

Alloy [for soldering aluminium]. P. A. J. H, Demeyer (F.P. 594,836, 6.3.25).—An alloy suitable for soldering aluminium to aluminium, iron, copper, lead, steel, brass, or bronze comprises 15—64% Zn and 85—36% Cd and is made by adding cadmium to molten zinc. A. R. Powell.

Aluminium alloys. G. Kroll (F.P. 594,851, 7.3.25. Conv., 8.3.24).—Alloys of aluminium with 20—35% Si and up to 5% of silver, titanium, zirconium, or boron or mixtures of these elements are claimed. The resulting alloys are much harder than those containing less silicon and also have a smaller coefficient of expansion. A. R. Powell.

Hard [lead] alloys. Materiel Téléphonique Soc. Anon. (F.P. 595,419, 3.2.25).—A mixture of lead with antimony, tin, lead, aluminium, or zinc is heated for a long time at a temperature below the m.p. of the mixture. The mass is then quickly cooled and aged at a temperature below 100°. For example, a mixture of 97·5 pts. of lead and 2·5 pts. of antimony is heated for 72 hrs. at 240°, cooled quickly, and aged; the resulting mass has a tensile strength of 770 kg. per sq. dm. A. R. Powell.

Treating sulphide ores [for recovery of sulphur, copper, and iron]. R. D. Pike (U.S.P. 1,570,777, 26.1.26. Appl., 13.6.23).—Sulphide concentrates containing copper and iron are heated with a solution of ferric chloride or sulphate in an autoclave fitted with a steam injector, the temperature being maintained above the m.p. of sulphur (*e.g.*, 140°). The residue is distilled for the recovery of sulphur and then worked up for the recovery of the precious metals. The solution is treated with pyrrhotite to reduce the excess of ferric salt used in leaching, then with sponge iron to precipitate copper and the precious metals. The residual ferrous chloride or sulphate solution is electrolysed to recover part of the iron and regenerate ferric chloride for use again in the process. A. R. Powell.

Protective coatings for metallic surfaces. E. Weintraub, and Soc. Alsacienne de Constructions Mécaniques (F.P. 592,986, 22.4.24).—Metallic surfaces may be protected from oxidation and rendered non-porous by a coating of aluminium borate or other refractory borate. Metal treated in this way does not evolve gas when heated to high temperatures or when subjected to a high vacuum and is therefore suitable for electric vacuum apparatus. A. R. Powell.

Electrolytic preparation of alkaline-earth metals. Chem. Fabr. Kalk, H. Oehme, and

E. HERRMUTH (G.P. 420,042, 24.11.23).—A contact cathode which is gradually raised is used to prevent re-melting of the deposited alkaline-earth metal; the upper part of the rod, above the level of the fused electrolyte, is cooled from outside by means of a stream of gas, or by means of a cylinder, through which water flows. R. B. CLARKE.

Melting light metals, *e.g.*, aluminium, magnesium, or their alloys in an induction furnace. RHEINISCHE METALLWAREN- U. MASCHINENFABR. (G.P. 421,281, 6.2.24).—Light metals and alloys are melted in an induction furnace in an atmosphere of an indifferent gas under such pressure that the combined pressure of gas and molten metal is sufficient to overcome the pinch effect in the secondary circuit. The furnace is enclosed in a gas-tight chamber provided with valves for the admittance of the compressed gas, the valves being coupled with the doors of the furnace so that the latter cannot be opened until the internal pressure has been relieved. A. R. POWELL.

Recovery of zinc from burnt pyrites after a chloridising roast. M. SCHMIDT (G.P. 422,044, 15.7.24).—The solutions obtained by leaching burnt pyrites that has been subjected to a chloridising roast are separated according to their content of sulphate and the solutions richest in sulphate are used for leaching fresh charges of roasted ore so as to enrich them in zinc and reduce the sulphate content. The enriched solutions are treated with calcium chloride to remove the sulphate as calcium sulphate and the weak zinc solutions are treated with milk of lime to obtain a mixture of zinc hydroxide and calcium sulphate. During the leaching process the ore is added to the solution as hot as possible. A. R. POWELL.

Electrolytic deposition of chromium. H. WOLFF (G.P. 422,461, 28.3.23).—An anode of pressed and sintered chromium powder is used in the electroplating of articles with chromium. This anode does not become passive and the bath is therefore not denuded of chromium and may be used for chromium-plating drawn wires. A. R. POWELL.

Aluminium alloy. R. L. JOHNSTON, R. S. ARCHER, and Z. JEFFRIES, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,572,489–90, 9.2.26. Appl., 27.11.20 and 11.2.25).—See E.P. 172,018; B., 1923, 361 A. Special claim is made for an alloy containing Si 8, Zn 10, and Cu 2·5%.

Production of alloyed steel and iron with chromium, manganese, etc. T. G. and F. E. L. STIG (U.S.P. 1,571,382, 2.2.26. Appl., 21.3.24).—See E.P. 213,568; B., 1925, 13.

Metal-extraction process. L. VENN-BROWN (U.S.P. 1,571,502, 2.2.26. Appl., 23.9.24).—See E.P. 239,720; B., 1925, 887.

Method of starting electrolytic cells [for refining metals]. D. H. TILSON, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1.572,253, 9.2.26. Appl., 30.11.23).—See E.P 225,494; B., 1925, 362.

Smelting or heating furnace with firing of coal dust. E. VOGT and L. KIRCHHOF (U.S.P. 1,572,336, 9.2.26. Appl., 26.3.25).—See E.P. 231,513; B., 1925, 867.

Making castings of aluminium-silicon alloys. R. S. ARCHER and J. D. EDWARDS, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,572,459, 9.2.26. Appl., 27.11.20).—See E.P. 171,997; B., 1923, 662 A.

Metallurgical furnaces. B. TALBOT (E.P. 246,627–8, 5.12.24).

Cupola furnaces with hot blast apparatus. E. VANDERSTEIN (E.P. 246,932, 11.11.24).

Annealing furnace (U.S.P. 1,569,356).—See I.

Treating alkali liquors from purification of lead (E.P. 245,479).—See VII.

Crucibles (U.S.P. 1,570,064).—See VIII.

XI.—ELECTROTECHNICS.

Hydrogen-ion control of nickel-plating baths with the quinhydrone electrode. H. C. PARKER and W. N. GREER (Trans. Amer. Electrochem. Soc., 1926, 49, 9—20. Advance copy).—The most satisfactory p_H for nickel-plating solutions is between 5·7 and 6 and this range is most conveniently determined by the use of a quinhydrone electrode. The apparatus consists of a portable potentiometer, a Parker and Dannerth combination cell (B., 1925, 555), a beaker, and a burette. About 200 c.c. of the solution to be tested are placed in the beaker, 10 c.c. of a saturated quinhydrone solution are added, the combination cell is lowered into the liquid, a potentiometer measurement taken after 2—3 min., and the p_H value read from a graph. A method is described for maintaining the acidity of the bath at the correct figure without weighing and without the use of standard solutions. The quinhydrone electrode method of determining the p_H of nickel-plating baths is more accurate than any colorimetric method, is easier to manipulate, and requires less elaborate apparatus. A. R. POWELL.

Determining silver, gold, and platinum in anode slimes. ECKERT.—See X.

PATENTS.

Electrolytic cells. BADISCHE ANILIN- U. SODA-FABR., Assees. of G. PFLEIDERER (G.P. 421,784, 13.11.23).—To avoid disturbances in the cell caused by leakage, it is enclosed in a container, inside which is a gas, *e.g.*, nitrogen, steam, or one of the gases produced by the electrolysis, under pressure. R. B. CLARKE.

Producing lead oxide [from storage-battery plates]. S. M. Evans (U.S.P. 1,570,438, 19.1.26. Appl., 5.3.23).—Finely divided lead compounds from storage-battery plates are treated with a hot solution of an alkali-metal base, washed, and oxidised by heat. J. Grant.

Exposure of liquids to ultra-violet rays. Quarzlampen-Ges. m.b.H. (G.P. 421,568, 26.5.23). —By centrifugal force the liquid is drawn in a thin layer up the inner walls of a revolving drum inside which is a source of ultra-violet light and which provides for a circulation of the liquid. By tubes leading down into the bulk of the liquid, air or other gases may be introduced. Bleaching and polymerisation of oils, softening of water, sterilisation, and acceleration of fermentation are among the suggested uses. S. S. Woolf.

Electrolysing fused baths. R. J. McNitt (E.P. 246,542, 30.10.24).—See U.S.P. 1,524,268; B., 1925, 249.

See also pages 224, **Electrical precipitation** (E.P. 246,046). 237, **Didymium and cerium** (F.P. 594,783). 245, **Thin wires** (E.P. 235,893); **Welding electrodes** (E.P. 230,831 and 245,635); **Protective coatings for metals** (F.P. 592,986).

XII.—FATS; OILS; WAXES.

Marine animal oils. Sperm whale oil and spermaceti. E. André and T. François (Compt. rend., 1926, **182**, 497—499).—Glycerol has been isolated from sperm whale oils, after saponification, in the following proportions:—Oil from the cranial cavity (sperm oil) 8·1%, subcutaneous fat 1·3%, oil from flesh 5·5%. Alcohols of high molecular weight occur to the extent of 36·4, 40·0, and 17·5% respectively in the same oils. Solid commercial spermaceti contains 0·7% of glycerol. L. F. Hewitt.

Fat from spent [hydrogenation] catalyst. C. Stiepel (Seifensieder-Ztg., 1925, **52**, 967—968; Chem. Zentr., 1926, I., 1486).—Considerable quantities (up to 1·8%) of a brownish viscous fatty mass were obtained when catalyst residues were saponified to recover the fat in them. The product thus obtained consisted of oxidised fatty acids with a low iodine value and high saponification value. As no such compounds were contained in the raw materials of the process, they must have been formed in the hydrogenation process. According to Normann, oxygen present in the hydrogen used could have this effect. S. Binning.

See also A., 239, **Interfacial tension and emulsification** (Harkins and Zollman). 241, **Solutions of sodium behenate** (Laing).

Drying of fatty oils. Eibner and Rasquin.—See XIII.

Patent.

Exposing liquids to ultra-violet rays. (G.P. 421,568).—See XI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Fastness of pigments to light. H. Wagner (Farben-Ztg., 1926, **31**, 1023—1026, 1073—1076).—The use of artificial light sources in the examination of pigments for permanency is discussed. Parallel tests on 75 coal tar colours and their lakes and 14 mineral pigments, negative the possibility of deducing the actual time of withstanding sunlight from the results of accelerated tests, the conversion factor of "ultra-violet lamp hours" into "sun hours" differing widely with the different pigments. With this limitation, however, exposure to the ultra-violet lamp is a test of great value, since the fading follows the same general course as on exposure to sunlight, and reaches the same end point where complete bleaching does not occur. For graphically representing the colour variations, the author uses ternary diagrams founded broadly on Ostwald's colour scheme (cf. B., 1919, 914 a). Factors affecting the fastness of a lake, *e.g.*, method of precipitation, adsorption on to the base, nature of the base, reduction with other pigments, refractive index and general nature of the medium, are considered from the experimental results and the optimum conditions inferred. The colours studied are finally arranged in 10 groups in descending order of fastness to light. S. S. Woolf.

Lead suboxide as pigment. A. V. Blom (Farbe u. Lack, 1925, 504—505; Chem. Zentr., 1926, I., 1005).—The gradual combination of lead suboxide with the decomposition products of linseed oil produces finely dispersed lead soaps which assist the rapid solidification of the film by opposing the swelling of the linoxyn, and also diminish the tendency of linoxyn to crumble on prolonged keeping, *e.g.*, for a year. S. S. Woolf.

Catalysis of the drying of fatty oils at ordinary temperatures; formation of varnish in the cold. A. Eibner and H. Rasquin (Chem. Umschau, 1926, 33, 29—38; cf. B., 1925, 930).—The authors stress the relativity of the widely accepted division of fatty oils into drying, semi-drying, and non-drying oils, and do not accept the statement that "the olein in linseed oil does not dry." The behaviour of a typical non-drying oil (olive oil) was observed alone and mixed with driers and pigments on glass, on lead, and over a red lead-poppyseed oil primer. In the last case a particularly marked acceleration of the drying resulted. The chemical reactions involved are identical with those of drying oils, and although the delay in the physical film-forming processes renders the oil unsuitable for paint and varnish, yet, owing to the gradual nature of the changes, the drying of such oils provides more valuable information than the drying of linseed oil, which has been investigated almost exclusively. A further possibility is opened up by the comparatively rapid air-drying of olive oil that has been exposed to ultra-violet light for about a week. A sample of Andalusian olive oil was found to have the composition: oleic acid 79·7, α-linoleic acid 6·7,

palmitic acid 5·5, stearic acid 3·4, glycerol residue 4·2, unsaponifiable matter 0·9%. The glycerides present were triolein, α-linoleodiolein, oleodipalmitin, and stearodiolein, the first-named being present in largest quantity and the sum of the first two being larger than the sum of the last two. S. S. WOOLF.

Composition of oils of Bucovinian fir trees. O. CZERNY (Bul. Soc. Chim. Romania, 1926, 7, 91—93; cf. A., 1924, i., 659; B., 1925, 179).—By the distillation of fir tree rosin a crude oil is obtained with a blue fluorescence and disagreeable smell. This contains abietic acid (m.p. 156°) which can be extracted by sodium carbonate solution. The purified oil gives the Storch–Morawski reaction (violet colour with benzene, acetic anhydride, and stannic chloride). After further purification with potassium hydroxide solution at 50—60°, a clear yellow odourless oil is obtained with no fluorescence. On fractional distillation under a pressure of 9 mm. the following substances are obtained:—(1) 195—198°, a colourless oil, d^{15} 0·9647, of empirical composition $C_{18}H_{26}$, which on oxidation and saponification yields trimellitic acid; (2) 240—265°, a colourless, odourless oil, d^{15} 0·960, consisting of styrene; (3) 310—340°, a light blue oil of composition C_5H_4; (4) 340—348°, a greenish aromatic oil, d^{15} 0·9686, which on extraction with sulphuric acid yields diterebenthyl (Renard, 1888, A., 161). The residue from the sulphuric acid extraction, when heated with sulphur as directed by Renard (*loc. cit.*), yields an oil, b.p. 392—394°, identified as retene. Corrections to an earlier paper (*loc. cit.*) are given.
W. HUME-ROTHERY.

Relation between the viscosity of resin solutions and the constitution of the solvent. E. KEYSSNER (Z. angew. Chem., 1926, 39, 99—105).—Using a given resin and different solvents in molecular proportions, the viscosity of the solutions is practically identical with solvents belonging to a homologous series. Almost identical viscosities are shown also by solutions in benzene homologues, in di-, tri-, and perchloroethylene, and in various petroleum products. The magnitude of the viscosity depends markedly in the constitution of the solvent, being much greater with saturated compounds, *e.g.*, per- and pentachloroethane, than with unsaturated compounds, *e.g.*, dichloroethylene. Differences in the behaviour of isomerides, *e.g.*, *o*- and *m*-xylene, are very slight. The presence of a hydroxyl group in the solvent appears to cause an increased viscosity.
W. T. K. BRAUNHOLTZ.

Aeroplane dopes. M. DESCHIENS (Chim. et Ind., 1926, 15, 17—32).—The author traces the historical development of cellulose ester dopes and describes the functions of their constituents (base, light solvents, diluents, heavy solvents, and plasticisers). The acetylation of cellulose is briefly discussed. Various formulæ for pigmented and non-pigmented dopes are given, and the different methods for their manufacture, the nature of the surface to be coated, and methods of application, mechanically or by hand, are described. Determinations of the rate of evaporation of solvents from first, second, and third coats showed that the first coat dries relatively slowly and requires a greater weight of dope owing to penetration of the fibre; the dried film retains appreciable amounts of heavy solvent for a period exceeding three months. The elasticity, impermeability, and tensile strength of fabrics are increased and air friction is lessened by doping.
S. S. WOOLF.

PATENTS.

Phosphorescent or luminous masses. H. M. MINES (E.P. 245,612, 18.5.25).—Phosphorescent materials of good durability and intensity that may be sensitised by artificial light and that will withstand grinding to pigment size without loss of luminosity, consist of a base composed of mixed alkali and alkaline-earth carbonates, with which is incorporated sulphur, small amounts of carbonaceous matter, such as starch, and very small amounts of "phosphorogens" and "luminophores," the whole being subjected to heat. The phosphorogens—thallium, thorium, uranium, bismuth, silver, or nickel compounds—act as light-emission centres, whilst the luminophores—sodium, potassium, manganese, barium, or calcium compounds—act as fluxes and convey the phosphorogens throughout the entire mass during the heat treatment. Formulæ for products emitting phosphorescence of the seven main spectrum colours are given as well as suggestions for obtaining intermediate colours. S. S. WOOLF.

Coumarone resin composition. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,570,584, 19.1.26. Appl., 12.11.17).—Coumarone resin containing acid substances is heated above its melting point, and simultaneously treated with a basic neutralising agent, in the absence of sufficient water to dissolve the basic material. B. W. CLARKE.

Preparation of solutions of organic substances such as cellulose esters, resins, dyes, caoutchouc, etc. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING, Assees. of R. LEOPOLD and A. MICHAEL (G.P. 419,223, 2.2.24).—The solvents consist of *cyclo*acetals, prepared from aldehydes and α- or β-glycols, either alone or mixed with additional substances, especially alcohols. The following examples are given: ethylene ethylidene ether, $Me\cdot CH<\begin{matrix}O-CH_2,\\ | \\ O-CH_2\end{matrix}$ b.p. 80—82°, from glycol and acetaldehyde; ethylene butylidene ether, $Pr^{\alpha}\cdot CH<\begin{matrix}O-CH_2,\\ | \\ O-CH_2\end{matrix}$ b.p. 130°, from butaldehyde and glycol; acetal of 1:3-butylene glycol,

$$\begin{matrix}MeCH\cdot CH_2\cdot CH_2\\ | \qquad\qquad | \\ O\cdot CHMe\cdot O\end{matrix}$$

b.p. 180°; product from formaldehyde and 1:3-butylene glycol, b.p. 115—120°. A. COULTHARD.

Preparation of a solvent which increases the drying capacity of drying oils, varnishes, etc. M. LUGEON (F.P. 592,595, 3.2.25).—Oil of turpentine is treated (warm) with a mixture of hydrogen and

carbon monoxide, preferably water-gas, in the presence of an acid, for example, oxalic acid. The product so obtained is then treated with compounds of manganese, lead, cobalt, or titanium, for example, manganese dioxide, and the resinates or linoleates of these metals. The solvent is freed from insoluble matter by decantation. A. Coulthard.

Reclaiming asphalt with production of lampblack. (U.S.P. 1,569,462).—See II.

Dyestuff emulsions. (E.P. 245,678).—See IV.

Exposing liquids to ultra-violet rays. (G.P. 421,568.)—See XI.

Treating pulverulent material. (U.S.P. 1,561,971.)—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Patents.

Treating pulverulent material [clay]. J. G. Coffin and A. W. Keen, Assrs. to Naugatuck Chemical Co. (U.S.P. 1,561,971, 17.11.25. Appl., 16.10.23.).—An aqueous suspension of clay is freed from coarse particles by settling, thickened by further settling, and then, while still fluid, is sprayed into the top of a chamber up which air mixed with hot furnace gases at a temperature of about 350° is passed. The clay is dried in its fall through the chamber and is obtained in the form of lightly cohering globular particles. The process is specially applicable to the preparation of clay for use as a filler for rubber goods and may also be applied to other fillers, pigments, etc., and to the preparation of clay mixtures in the ceramic industry, different clays being mixed together in the form of slips and the mixture dried as described. T. S. Wheeler.

Solutions of caoutchouc (G.P. 419,223).—See XIII.

XV.—LEATHER; GLUE.

Reversal of the double refraction of collagen fibres when treated with certain vegetable tannins. A. Kuntzel (Collegium, 1925, 623—627; cf. Ebner, A., 1896, ii., 457).—The plane of polarisation in which light is polarised by collagen fibres is rotated through 90° (*i.e.*, the double refraction is changed from positive to negative) when the latter are treated with gallotannic acid, sumach, tea, divi-divi, algarobilla, or myrobalans. Those tannins which do not change the plane of polarisation, cause weakening of the original double refraction of the collagen fibres. The phenomenon can be used for ascertaining the tannage used on a leather. Synthetic tans and sulphite-cellulose have no effect on the double refraction of the collagen fibres.
D. Woodroffe.

Behaviour of sharpened limes in unhairing. M. Kaye and R. H. Marriott (J. Soc. Leather Trades Chem., 1925, **9**, 591—620).—Pieces of dried goatskin, fresh ox hide, or calfskin were treated with various solutions. Lime liquor attacked and partially dissolved the cells of the hair roots and follicles until the latter collapsed away from the hairs. The lowest layer of the epidermis was also attacked and the epidermis split off completely in 7—8 days. The unhairing power of the sulphides of sodium, potassium, barium, and calcium is practically the same. Sulphides of ammonium and magnesium do not unhair. Bivalent sulphide ions are necessary for unhairing. Hydrosulphide ions do not unhair. The presence of hydroxyl or hydrosulphide ions does not prevent unhairing if bivalent sulphur ions are present. Polysulphides unhair similarly to sulphides. Their unhairing effect increases with the concentration and with the addition of hydroxyl ions, and decreases with the complexity of the polysulphide. Previous soaking of skin in lime or caustic soda liquors retards unhairing with sulphides or polysulphides according to the time of soaking and the concentration of the soak liquor. Acid soaking does not affect the unhairing. The rotting of the hair shaft is not a true measure of the progress of unhairing. Sodium sulphide dissolves hair due to a reaction between its sulphur and the sulphur of the hair. The cystine of hair is removed when the hair is treated with caustic soda solutions, hence previous alkaline soaking of skin alters the hair and the sulphide cannot combine with it. In sulphide liquors there is a reaction between soluble nitrogen products, sulphide sulphur, and oxygen from the air, resulting in a rapid disappearance of the sulphide sulphur. The presence of a film of grease on the hair does not prevent combination with sulphide sulphur, but protects the hair from the solvent action of the sulphide liquor. The action of lime on hair leads to the formation of sulphides in the liquors.
D. Woodroffe.

Patent.

Tans comprising a reaction product of sulphite-cellulose. J. Breedis, Assr. to Rohm and Haas Co. (U.S.P. 1,563,010, 24.11.25. Appl. 23.2.24).—Natural tans, partially insoluble in water, are converted into water-soluble tans by combining them with sulphite-cellulose waste lye by heating in the presence of free alkali, and then slightly acidifying the mixture. R. B. Clarke.

XVI.—AGRICULTURE.

Nitrogen recuperation in the soils of the Bombay Deccan. I. D. L. Sahasrabuddhe and J. A. Daji (Mem. Dep. Agric. India, 1925, **8**, 53—68). —The influence of various conditions on fixation of nitrogen in a Deccan soil taken at the end of the hot weather has been studied. On addition of water, fixation of nitrogen begins rapidly and continues for about 35 days. The amount fixed increases with addition of water up to 30%. Drying and re-moistening after fixation has taken place still further increases the amount of nitrogen fixed. Both fixation and nitrification are greater at 40° than at lower temperatures. C. T. Gimingham.

Better utilisation of phosphoric acid present in soils. J. WITYN (Z. Pflanzenkrankh., 1925, A 6, 27—51; Chem. Zentr., 1926, I., 1019).—The influence of the reaction of the soil on the nature of the phosphoric acid compounds present is discussed. Plants suffer more from lack of phosphorus in acid soils than in similar soils when the acidity has been removed by liming. On strongly acid soils, an application of mineral phosphates before liming is recommended.

C. T. GIMINGHAM.

Neubauer's "seedling" method [for determining manurial requirements of soils]. E. GÜNTHER (Z. Pflanz. Düng., 1926, B 5, 32—36).—Critical experiments on Neubauer's method for determining the phosphoric acid and potassium requirements of soils show that considerable variations in the light intensity during growth of the seedlings affect the percentage of nutrients absorbed to a small extent only. Similarly, the absorption of nutrients by the seedlings is almost unaffected by changing the p_H of a soil from an initial value of 5·0 to 6·5 and 8·0 by addition of calcium carbonate. Under normal conditions, both light intensity and soil reaction can be neglected in carrying out the test.

C. T. GIMINGHAM.

Comparative fertilising activities of different forms of nitrogen in new urea fertilisers derived from cyanamide. C. BRIOUX and J. PIEN (Compt. rend., 1926, 182, 410—412).—Addition of ammonium sulphate to soils increases nitrification, guanylurea sulphate has a slight depressing action, whilst dicyanodiamide almost completely suppresses nitrification. The following proportions of the nitrogen administered were absorbed by maize and white mustard plants when treated with the fertilisers named: sodium nitrate 70·3%, ammonium sulphate 68%, cyanamide 64%, urea 63·1%, urea sulphate and phosphate 64%, "phosphazote" (containing 25% of the nitrogen present as guanylurea sulphate) 45·9%, guanylurea sulphate 13·2%, dicyanodiamide 6%. Since guanylurea sulphate is practically inactive, and dicyanodiamide toxic to the plant, the proportions of these in artificial manures must be maintained at a minimum.

L. F. HEWITT.

Determination of phosphoric acid as magnesium ammonium phosphate. G. JÖRGENSEN (Analyst, 1926, 51, 61—72).—Several methods that have been proposed for the determination of phosphoric acid in mineral phosphates and fertilisers have been examined and the sources of error are pointed out (cf. A., 1925, ii., 824). The following procedure, which has been tested over a period of several years, is recommended to give results correct to within 0·1%. Three stock solutions are prepared: (*a*) 100 g. of ammonium molybdate are dissolved in 280 c.c. of ammonia (*d* 0·97), 300 c.c. of this solution are poured into 700 c.c. of nitric acid (*d* 1·21), and the mixture is filtered on the next day; (*b*) 80 c.c. of ammonia (*d* 0·91) are added to a mixture of 90 c.c. of nitric acid (*d* 1·4) and 1500 c.c. of water, and the solution is diluted to 2 litres; (*c*) 50 g. of magnesium chloride and 150 g. of ammonium chloride are dissolved in water and the solution is diluted to 1 litre. For the analysis of mineral phosphates, 5 g. are dissolved in 25 c.c. of nitric acid (*d* 1·21) and 12·5 c.c. of hydrochloric acid (*d* 1·12), the solution is diluted to 250 c.c. and filtered and 50 c.c. of the filtrate are treated with the requisite amount of (*a*). The mixture is heated for 10 min. at 50° on a water-bath, cooled, and filtered, the precipitate is washed ten times with 20 c.c. of (*b*) and dissolved in 100 c.c. of 2·5% ammonia solution, and the solution is treated at 100° with 30—35 c.c. of (*c*) added drop by drop with stirring. After cooling for 4 hrs. the precipitate is collected and converted into magnesium pyrophosphate as usual. For the determination of the total phosphoric acid in Thomas slag, 5 g. are dissolved in 40 c.c. of hydrochloric acid (*d* 1·18) without addition of nitric acid. Evaporation to render the silica insoluble is unnecessary; it may in some cases result in loss of phosphoric acid. The remainder of the analysis is conducted as described above. The quantity of solution (*a*) to be used is computed roughly from the equations $x=0·12y+345p$ for mineral phosphates and superphosphates and $x=0·34y+412p$ for Thomas slag, where x is the number of c.c. of (*a*), y is the volume of the phosphate solution, and p the content of phosphoric anhydride. Direct precipitation of magnesium ammonium phosphate from ammoniacal solutions of commercial phosphates in the presence of ammonium citrate yields high results if considerable quantities of sulphates, ferric, aluminium, or calcium salts, or any silica or arsenic acid are present.

A. R. POWELL.

Determination of ammonia in fertilisers by the formaldehyde method. W. SELKE (Chem.-Ztg., 1926, 50, 83).—For the determination of ammonia in comparatively pure ammonium chloride or sulphate the formaldehyde method gives good results if the solution is made neutral to phenolphthalein before adding the formaldehyde. With ammonium superphosphate or crude preparations of the sulphate complications are introduced by the presence of acidic impurities, such as the salts of iron and aluminium. In the case of the phosphate these salts produce a turbidity on neutralising and the end-point is difficult to discern; addition of sodium citrate overcomes this difficulty but even then good results are obtained only when a small weight of salt (say 0·5 g.) is taken. The concentration of the solution after the addition of the formaldehyde has an appreciable effect on the results. To correct for this a solution of ammonium sulphate containing approximately the same amount of ammonia in the same volume as the solution tested is prepared and titrated with alkali until phenolphthalein is just coloured pink; a few drops of methyl orange are added and the solution is titrated with acid until pink. The amount of acid so found is added to the c.c. of alkali used in the analysis proper.

A. R. POWELL.

Action of calcium and magnesium on seedlings of yellow lupin [*Lupinus luteus*]. BURK (Z. Pflanz.

Düng., 1926, B5, 1—23).—A detailed series of experiments in sand culture are recorded on the effect of calcium and magnesium chlorides, separately and together, and in presence and absence of potassium sulphate, on the early stages of development of yellow lupins. Calcium chloride was added at the rates of 1·58 g. and 4·74 g. per pot holding 1·5 kg. of sand, and magnesium chloride in equivalent amounts. In general, the presence of calcium chloride was harmful and of magnesium chloride beneficial; the difference in the effects of the two salts was particularly marked in regard to the development of roots and root-hairs. When magnesium chloride or potassium sulphate was added with calcium chloride, the injurious action of the latter was almost completely neutralised. The harmful effects of calcium chloride and the beneficial effects of magnesium chloride were due to the cations and not to the chlorine ions. The literature on the subject of the sensitivity of lupins to the presence of calcium is reviewed.

C. T. GIMINGHAM.

Mineral content of pasture grass and its effect on herbivora. W. ELLIOT, J. B. ORR, and T. B. WOOD. **I. General.** W. ELLIOT. **II. Effect of addition of mineral salts on the ration of sheep.** W. ELLIOT and A. CRICHTON. **III. Analyses of samples of British pastures.** W. GODDEN. **IV. Seasonal variations in mineral content of pastures.** (MISS) E. M. CRUIKSHANK. **V. Effect of fertilisers on mineral content of soils.** W. GODDEN (J. Agric. Sci., 1926, 16, 59—64, 65—77, 78—88, 89—97, 98—104).—I. There is no marked difference between the energy values of the herbage of good and poor pastures. Important differences occur in mineral composition, a high mineral content being associated with high nutritive value. Certain cases of excessive mortality in sheep are related to low mineral content of the pasture herbage. II. "Bent leg" in sheep is correlated with mineral deficiency in the food and with a general lower nutrition. Mineral deficiency in pasture herbage can be correlated with high mortality in sheep. III. The herbage of hill pastures is generally poorer than that of cultivated pastures in silica-free ash and in the individual constituents with the exception of sodium. It is also rather poorer in nitrogen. Herbage left ungrazed bears a similar relationship to grazed herbage. There are no marked differences in energy value as measured by calorimetry. Animals appear to choose herbage of high mineral content in grazing. IV. There is a definite seasonal variation, more marked in good than in inferior types, in the mineral content of pasture grasses. The variation is most pronounced in the case of calcium oxide, which attains a maximum and then decreases. Similar but smaller variations are shown by silica-free ash, sodium oxide, phosphoric oxide, chlorine, and nitrogen. The date at which the content of ash and of the individual constituents reach a maximum varies for different fields and is probably influenced by grazing. V. Considerable modifications are produced in the mineral composition of pasture herbage by the application of artificial fertilisers. The constituents showing the biggest variations are calcium, potassium, and, to a less extent, phosphorus. Increase in calcium is generally correlated with increase in nitrogen.

G. W. ROBINSON.

PATENTS.

Production of solid urea from solutions. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 245,687, 14.8.25).—A hot highly concentrated solution of urea is distributed in the form of a moderately coarse spray (not atomised). The drops solidify and urea is obtained in the form of "sandy" grains suitable for use as a fertiliser. If a 95% solution is used, no subsequent drying is required.

C. T. GIMINGHAM.

Decomposition of materials containing phosphoric acid. N. KRAUTZ and L. DE MOLTKE-HUITFELDT (F.P. 595,133, 10.3.25. Conv., 11.3 and 22.4.24).—Mineral phosphate, in large pieces, is treated with hydrochloric acid in towers.

C. T. GIMINGHAM.

Manufacture of salts of urea. J. BRESLAUER and C. GOUDET, Assrs. to SOC. D'ETUDES CHIM. POUR L'IND. (U.S.P. 1,572,638, 9.2.26. Appl., 2.5.22).—See E.P. 179,544; B., 1923, 901 A.

XVII.—SUGARS; STARCHES; GUMS.

Polarimetric determination of sucrose and its mixtures with other sugars. FINCKE.—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Partial pressures of aqueous ethyl alcohol. DOBSON.—See A., Mar., 235.

PATENTS.

Producing wort. AKTIEBOLAGET SEPARATOR (E.P. 233,321, 30.3.25. Conv., 5.5.24).—The slime separated from the primary wort is leached and the wort thus produced separated by centrifuging, this process being repeated on the slime from the secondary wort. The wort obtained at a subsequent centrifuging and freed from solid impurities is used as a leaching liquid for a slime obtained at a previous centrifuging.

T. H. POPE.

Preparation and use of lactates [in brewing etc.]. H. N. MURPHY (E.P. 246,278, 18.12.24).—Dry preparations containing lactates, for adding to water to be used in the manufacture of, *e.g.*, beer, vinegar, and malt extract, are obtained by adding suitable salts, such as calcium chloride, sodium sulphate, potassium chloride, and sodium phosphate, with or without the addition of soluble peptones, to lactic acid solutions, which may or may not be heated, and wholly or partially neutralising the acid by the addition of calcium oxide, hydroxide, or carbonate. On keeping, the solution sets to a homogeneous mass. For example, a product obtained by using 25% of lactic acid, 25% of water, 20% of calcium sulphate, 10% of potassium chloride, 5% of sodium chloride, 5% of magnesium sulphate, 3% of

peptone, and 7% of calcium oxide, is added to brewing water at the rate of ¼—1 lb. per 36 gals.

L. A. COLES.

Manufacture of lactic acid. S. E. FAITHFULL (U.S.P. 1,569,221, 12.1.26. Appl., 8.5.24).—In making lactic acid by the fermentation, in presence of lactic acid bacteria, at 44·5—45·5°, of an acid liquor which contains somewhat hydrolysed carbohydrate material and in which accumulation of excessive acidity is prevented, a gaseous oxidising material containing free oxygen is introduced in amount sufficient to retard considerably the formation of butyric acid. The liquor is afterwards made alkaline and heated to near the b.p. of water to convert the remaining carbohydrates into caramel-like products. The hydrogen-ion concentration is then adjusted and proteins and similar nitrogenous substances are removed. T. H. POPE.

Manufacture of aldehydes, oils, and organic oils from cacti. W. M. SINCLAIR (U.S.P. 1,569,339, 12.1.26. Appl., 25.2.25).—Cacti are mashed without water in conditions favourable to fermentation by their indigenous fungus; the fully fermented pulp is hydrolysed and then distilled, the aldehyde being condensed and the essential oil separated from the aldehyde. T. H. POPE.

Combustible materials from peat (G.P. 421,734).—See II.

Exposing liquids to ultra-violet rays (G.P. 421,568).—See XI.

XIX.—FOODS.

Effect of short periods of cold storage on beef and mutton. W. M. CLIFFORD (Biochem. J., 1925, 19, 998—1003).—Beef or mutton kept at −4° and 2° are identical in appearance with freshly killed meat up to the third day of storage. The meat kept at −4° shows ice spicules and the red colour characteristic of frozen meat on the sixth day of storage. In hot English weather beef and mutton will not keep for 6 days in a room at 2°. There is no change in total nitrogen, soluble nitrogen, amino-nitrogen, carnosine, or creatine in meat kept at 2° for 3 days or at −4° for 13 days.

S. S. ZILVA.

Milk-fat determination in foodstuffs. J. KUHLMANN and J. GROSSFELD (Z. Unters. Nahr. Genussm., 1925, 50, 329—346).—Experiments have shown that the Reichert–Meissl values determined on small quantities of milk-fat and calculated to 5 g. are too high. The effect is even more pronounced in the case of coconut oil, though the presence of other fats may partially compensate for it. The difference (saponification value — Reichert–Meissl value — 200) is approximately proportional to the content of coconut oil in a mixture, and may be used to calculate a correction to be applied to the Reichert–Meissl value when coconut oil is present. The application of the A- and B-values of Bertram, Bos, and Verhage to the determination of coconut oil and milk fats (cf. B., 1926, 140) is discussed, and tables are given showing their values for various fat-contents. Other tables show the extent of the Reichert–Meissl value error for various quantities of milk-fat and coconut oil, and the content of milk-fat in mixtures corresponding to different Reichert–Meissl values, taking an average figure of 27 for milk-fat. J. GRANT.

Examination of confectionery products containing sugar and milk or cream. J. KUHLMANN and J. GROSSFELD (Z. Unters. Nahr. Genussm., 1925, 50, 346—351; cf. preceding abstract).—Tables are given showing the compositions of a number of commercial products, and the procedure for the analysis of such products is discussed. Fat content is determined by treating an aqueous solution of the sample with copper sulphate and sodium hydroxide solution, filtering off the coagulum, drying it, and extracting with a fat solvent.

J. GRANT.

Polarimetric determination of sucrose and its mixtures with lactose and other sugars, in cacao products, condensed milk, and sugar products. H. FINCKE (Z. Unters. Nahr. Genussm., 1925, 50, 351—365).—In the polarimetric determination of sucrose in cacao products, a factor has to be introduced to correct the reading for the volume of the insoluble matter. The values of this factor (x) are given in tables for a number of readings; or the following formula may be used:— $x=1-(0{\cdot}1-0{\cdot}0072d)r$, where d is the observed reading and r the volume of 1 g. of insoluble matter (0·9 c.c. for chocolate, and 0·7 c.c. for cocoa–sugar mixtures). The solutions are prepared for the polarimeter by the usual precipitation with lead acetate, but for milk-chocolate and condensed milk a second polarimeter reading must be taken after treatment with lime water (cf. B., 1924, 489). Tables are given for the determination of lactose from the difference of these readings, and correcting factors for the presence of lævulose, dextrose, maltose, and invert sugar have also been determined.

J. GRANT.

Amyl alcohol unsuitable for milk testing by Gerber's method. S. GOY and J. JANISCH (Z. Unters. Nahr. Genussm., 1925, 50, 373).—When used for Gerber's test on milk, two samples of amyl alcohol were found to give results 0·4 and 0·6% too high. Blank tests using water in place of milk resulted in the separation of an unsaponifiable oil resembling petroleum. J. GRANT.

Variations in mineral content of cabbage and sauerkraut. W. H. PETERSON, C. A. ELVEHJEM, and L. A. JAMISON (Soil Sci., 1925, 20, 451—457).—Analyses of samples of cabbage and of sauerkraut from different localities showed wide variations in the percentages of calcium, phosphorus, iron, sugar, and nitrogen. There is a loss of 10—20% of the mineral constituents in making sauerkraut, if the amounts present in the juice are neglected.

C. T. GIMINGHAM.

Mineral content of pasture grass. ELLIOT, ORR, and WOOD.—See XVI.

PATENTS.

Apparatus for drying casein curd. E. FOSTER, Assr. to VERMONT CASEIN PTY., LTD. (U.S.P. 1,571,518, 2.2.26. Appl., 12.12.22).—See E.P. 198,957; B., 1923, 852 A.

Preparation of aerated beverages. O. A. ELIAS (U.S.P. 1,572,111, 9.2.26. Appl., 20.3.23).—See E.P. 197,499; B., 1923, 676 A.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of nicotine by the method of Ulex. F. MACH and F. SINDLINGER (Z. anal. Chem., 1926, 67, 369—386).—The method described by Ulex for the determination of nicotine (B., 1911, 237) is shown to be erroneous at every stage of the operations. The loss of nicotine during the various treatments may amount to 9—26% of the total, 4—5% of the ammonia present in the material fails to be removed, and a quantity of ammonia equal to 20—25% of that originally present is produced by decomposition of nitrogenous compounds during the analysis. The total amount of ammonia that is finally titrated with the nicotine exceeds by 1—2% the nicotine lost by evaporation and decomposition, so that by this process it is possible to find a value for nicotine in plants that do not contain this alkaloid. The only exact method for the determination of nicotine is that involving precipitation with silicotungstic acid of the distillate obtained by distilling the substance with alkali in a current of steam. A. R. POWELL.

Nopinene as a technical raw material. G. AUSTERWEIL (Chem.-Ztg., 1926, 50, 5—6, 33—35; cf. B., 1925, 613).—The physical properties of pinene (m.p. −57°, b.p. 154·5—155°, d^{15} 0·8595, n_D^{12} 1·166, dispersion Hg yellow / Hg green 1·29) are contrasted with those of nopinene (m.p. −61°, b.p. 162—163°, d^{15} 0·8635, n_D^{12} 1·487, dispersion Hg yellow / Hg green 1·06). A method for the technical separation of these substances is based on the greater solubility of nopinene in organic solvents miscible with water, and a table showing the solubilities in alcohol containing varying amounts of water is given: 60% aqueous alcohol dissolves almost exclusively the nopinene from a mixture of the terpenes. A greater yield of bornyl chloride is obtained from nopinene than from rectified turpentine oil, by the action of hydrogen chloride, but further cooling is necessary. Bornyl esters are obtained more readily from nopinene than from pinene and, more important, with careful attention to temperature and acid concentration, contamination with monocyclic terpenes can be avoided. L. M. CLARK.

PATENTS.

Extraction of the essential principles of plants, flowers, fruit, and the like. E. FORAY (E.P. 232,552, 9.1.25. Conv., 19.4.24).—Perfumes and similar essences are extracted by means of ether vapour, and are subsequently absorbed from the vapour by means of sago, flour, sugar, alcohol, or similar material. L. A. COLES.

Manufacture of compounds of alkaloids. A. G. BLOXAM. From HACO-GES. A.-G. (E.P. 245,838, 14.10.24).—Stable compounds insoluble in water, and of therapeutic value, are produced by bringing together a nuclein or a nucleic acid, or compounds containing them, and an alkaloid, and subsequently coagulating the product, *e.g.*, by heat. For example, a solution of 2 kg. of morphine hydrochloride is added to a suspension of 100 kg. of yeast in 500 litres of water, and, after maintaining the solution at 60—65° for ½ hr., it is brought to the boil by introducing steam, and the coagulated product is removed and dried. L. A. COLES.

Producing chloro-derivatives of methane. H. WADE. From S. KARPEN AND BROS. (E.P. 245,991, 27.4.25).—Mixtures of 10—30% by vol. of chlorine with 90—70% of methane and methyl chloride are passed through a reaction chamber maintained at 350—800°, but preferably at 400—500°, and the products pass successively through a scrubber for removing hydrogen chloride, a gas-holder, a compressor in which they are compressed to 10—15 atm., a condenser, and thence into apparatus for separating the condensed liquid, which consists of a mixture of methylene chloride, chloroform, and carbon tetrachloride, from residual gas. The liquid is withdrawn and separated into its constituents by fractional distillation, and the gas is mixed with fresh supplies of methane and chlorine and returned to the reaction chamber. In operating the process to obtain a yield of about 80% of methylene chloride, 15% of chloroform, and 5% of carbon tetrachloride, 253·75 vols. of residual gas, containing about 60% of methane and 40% of methyl chloride, are mixed with 100 vols. of chlorine and 46·25 vols. of methane, before re-entry into the reaction chamber.
L. A. COLES.

Lactates (E.P. 246,278).—See XVIII.

Lactic acid (U.S.P. 1,569,221).—See XVIII.

Aldehydes, oils, and acids from cacti (U.S.P. 1,569,339).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Fogging action of hydrogen peroxide. W. CLARK (Phot. J., 1926, 66, 78—84).—Examination of the fogging action of hydrogen peroxide on single-layer plates prepared with a particular emulsion showed that the percentage of grains made developable increased with increasing time of treatment up to a maximum at about 60% and then decreased with further treatment. The larger grains in the emulsion were on the average more sensitive to peroxide than the smaller grains, the relative sensi-

tivities of different size classes being roughly parallel with their sensitivities to light. These facts are not in agreement with observations of Wightman, Trivelli, and Sheppard using another emulsion (J. Franklin Inst., 1925, 200, 335). In the case of a single-layer emulsion treated vertically in still hydrogen peroxide solution, the percentage of grains made developable in a given time falls off with increasing depth in the solution. The chemi-luminescence theory of the action of peroxide was examined by studying the effect of bathing normal plates in catalase solution on the photographic action of peroxide vapour and solution. The effect was always less with the catalase-bathed plates than with those bathed only in plain water, which is contradictory to what would be expected if the chemi-luminescence theory held, and seems to indicate a purely chemical action. The author does not now hold the view (cf. B., 1924, 116) that the sensitive centres in emulsion grains consist of silver oxide or hydroxide. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Modifying the brisance of explosives [azides]. L. BIRCKENBACH and W. RÖRIG (Preuss. Bergakad. Clausthal, Festschrift, 1925, 123—135; Chem. Zentr., 1926, I., 1492).—An investigation of the modification of the brisance of metal azides when they form mixed crystals or double salts. Double salt formation was detected in the systems lead azide–lead chloride and lead azide–lead bromide. The double salt with lead bromide was not exploded by a 10-kg. hammer falling through 100 cm., whereas the lead chloride double salt exploded when the same hammer fell 65 cm., and pure lead azide exploded when a 2-kg. hammer fell 35—40 cm. Both these double salts contain about 50% of the respective halide. The crystals of the chloride double salt are clear, transparent, long monoclinic needles with $a:b:c=1\cdot2347:1:1\cdot7179$ and $\beta=101°\ 4'$. S. BINNING.

Detection of carbon monoxide in after-damp. WEIN.—See II.

PATENTS.

Explosives [of the Sprengel type]. A. C. SCOTT and H. L. SULMAN (E.P. 246,352, 4.4.25. Addn. to 231,541; cf. B., 1925, 479).—Finely divided crystals of potassium chlorate are filled gradually into a cartridge case under pressure so that the volume of the voids between the crystals is less than 32% of the total volume occupied by the crystals in the cartridge. The cartridges are then impregnated with kerosene by partial immersion and upward percolation. S. BINNING.

Preparation of initiators [primers, caps, and detonators]. C. CLAESSEN (G.P. 419,556, 8.3.25).—Acetylacetone peroxide is used as either a main or a secondary charge or as a component. It is not particularly susceptible to shock or friction, but is easily caused to detonate by a spark or flame. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Rapid fine sand [water] filtration. H. W. BLAISDELL (J. Amer. Water Works' Assoc., 1925, 14, 581—597).—To operate a sand filter at high rates of flow the sand must be either dry when put into the filter or consolidated in place by agitation under water. By the combined use of fine sand, reverse flow, and filter washer, waters of medium turbidities, subject to occasional flood turbidities of 300 p.p.m., without settling, can be filtered at rates of flow of 25—50 million gals. per day, and waters of low-turbidity at a rate of flow of 50 million gals. per day. A high-percentage removal of bacteria, turbidity, and colour is effected. Efficiency depends upon the very fine surface sand, about $\frac{1}{8}$in. in thickness, left after washing. A suitable sand when screened to 20-mesh has a sufficient percentage of fines to accomplish the desired result. Reverse flow is permitted at the slow rate of 15 million gals. per day or $\frac{3}{8}$in. rise per min. for 5—15 min. daily, or oftener if required; no loss of filtered water is occasioned by this treatment. The quantity of raw water used for washing is 0·025% of the water filtered. All waters so far dealt with have been successfully treated without the use of alum. The cost of installation and operation of a rapid fine sand filter is less than one-half that of any other filter now in use. A filter-washer designed by the author is described. W. T. LOCKETT.

PATENTS.

Process for treating alumino-silicates [for use in softening water]. H. and H. REINBOLD (U.S.P. 1,570,006, 19.1.26. Appl., 21.1.24).—Material for softening water is produced from alumino-silicates, such as bentonite, by calcining the mineral after it has been ground and sized, and then treating it with a soluble aluminium compound, after which it is dried to remove excess water. L. A. COLES.

Oxygen from peroxides [for respirators]. HANSEATISCHE APPARATEBAU GES., VORM. L. VON BREMEN U. CO. (G.P. 419,610, 24.5.18).—Mixtures capable of suddenly liberating oxygen (*e.g.*, for respirators) are obtained by mixing an anhydrous alkali peroxide with an approximately equivalent amount of an anhydrous, difficultly volatile, acid anhydride or material liberating water of crystallisation or hydration only above 150°. Suitable substances are silica, tungsten trioxide, molybdenum trioxide, manganese dioxide, technically anhydrous copper sulphate, and alkaline-earth hydroxides. Substances, such as catalysts or acid-producing agents, which react with the above with evolution of heat are further appropriately added. A. COUSEN.

Filtering apparatus [for water]. W. PATERSON (U.S.P. 1,572,076, 9.2.26. Appl., 4.2.25).—See E.P. 233,031; B., 1925, 565.

Exposing liquids to ultra-violet rays (G.P. 421,568).—See XI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 16, 1926.

I.—GENERAL; PLANT; MACHINERY.

Device for circulating fluids under high pressure. I. R. McHaffie (Faraday Soc., Feb., 1926. Advance proof).—A small soft iron piston rod operating in a gun-metal cylinder is controlled electro-magnetically by two coils wound in the same direction and connected in series with a resistance so that they pass a current of 1 ampère. The coils are arranged so that they may be alternately short-circuited by a motor-driven commutator consisting of a brass slip ring in contact with a brass quadrant; thus the position of the magnetic field, and therefore that of the piston, is controlled by the rotation of the commutator. With a speed of 2 revolutions per second, the pump will circulate 25 litres of air per hour at atmospheric pressure; it can be operated as a lift pump or a force pump and circulates air up to 100 atm. pressure.

B. W. Clarke.

Absorption of chemical fogs. H. Remy (Z. angew. Chem., 1926, 39, 147—150; cf. B., 1925, 40).—Differences in the extent to which dry and moist fogs are absorbed by various agents are due to the particles of the moist fog being much larger than those of the dry. Thus, filter paper retains the moist fog better than the dry, whilst with active charcoal the reverse is true. Water converts a dry into a moist fog, the enlarged fog particles becoming thereby less mobile and less easily absorbed. Increasing the viscosity of a liquid absorbent diminishes its absorptive capacity by increasing the size of the gas bubbles passing through it, and so diminishing the chance of contact between liquid and fog particles.

W. T. K. Braunholtz.

Production of hydrogen by steam in a hot boiler tube. J. Porter (J. Roy. Tech. Coll. Glasgow, 1925, [2], 14—18).—No hydrogen could be detected in steam from a boiler tube when the latter was maintained at a temperature below 400°. Between 400° and 500° a very slow decomposition of the steam took place, which became very rapid at temperatures above 500°. A. R. Powell.

Fire-bars and their behaviour in the fire. R. Stumper (Chaleur et Ind., 1925, 6, 549—554; Chem. Zentr., 1926, I., 1277).—The life of fire-bars depends on the metallographic structure of the metal, the chemical composition of the fuel, the method of firing, and the boiler system. Phosphorus lowers the m.p. of the iron and renders it brittle at high temperatures; the presence of phosphates in the ash of the fuel causes it to sinter to the fire-bars and increases the possibility of a reaction between the ash and the metal of the bars with the formation of the ternary eutectic, steadite. The most harmful element in the fuel itself is sulphur. Grey cast-iron is less suitable than white cast-iron for the manufacture of fire-bars, as the presence of free graphite in the grey iron renders the metal porous, and air and deleterious gases can thus readily penetrate into the interior. White cast-iron resists the chemical action of the fire better than the grey variety and, although it is possible to cause decomposition of the cementite to take place, the carbon deposited does not cause the metal to become as porous as grey cast-iron.

A. R. Powell.

See also A., Mar., 217, **Critical potentials of hydrogen in presence of catalytic nickel and copper** (Wolfenden). 230, **Plasticity of amorphous and crystalline solids** (Becker). 240, **Mechanism of ultrafiltration** (Duclaux and Errera). 250, **Catalytic activity** (Constable); **Catalysis** (Zelinski).

Conditioning of boiler water. Hall.—See XXIII.

Patents.

Apparatus for purifying, cooling, heating, mixing, or absorbing gases and vapours. E. Theisen (E.P. 234,109, 13.5.25. Conv., 15.5.24). —The gas or vapour traverses one or more curtains of liquid discharged outwards by one or more vane wheels carried by a rotary shaft within a casing. Each wheel comprises a flat disc carrying, near the rim only, a series of oblique vanes, and the liquid is delivered to the central portions of these discs. Means may be provided for returning the liquid, discharged outwardly from one wheel, to the centre of the succeeding wheel. H. Holmes.

Centrifugal separators for separating solids from liquids. Laughlin Filter Corp., Assees. of W. C. Laughlin (E.P. 235,916, 22.6.25. Conv., 21.6.24).—A centrigugal separator has the separating zone divided into two parts by a vertical partition, on one side of which clear liquor is discharged, whilst on the other side, scrapers geared to rotate at a speed slightly different from the bowl, remove the deposited mud to a discharge outlet. The axial feed inlet is on the same side of the partition as the discharge outlet. B. M. Venables.

Dehydrating liquids. A. S. Elsenbast, Assr. to Celite Co. (U.S.P. 1,570,626, 26.1.26. Appl., 14.3.22).—Small quantities of water are separated from non-aqueous liquids by admixture of dehydrated diatomaceous earth, followed by filtration.

H. Holmes.

Heat exchanger. B. M. JOHNSON, Assr. to CARBORUNDUM Co. (U.S.P. 1,570,674, 26.1.26. Appl., 9.2.24).—Banks of conducting tubes are arranged side by side above a silicon carbide radiating wall of a combustion chamber, each bank comprising metal tubes arranged above silicon carbide tubes adjacent to the wall. Means are provided for so directing the combustion gases into contact with the tubes as to subject the carbide tubes of each bank to a higher temperature than the metal tubes, and a mixing chamber is disposed between the discharge ends of the tubes of each bank and the intake ends of the tubes of the next bank.

H. HOLMES.

Treating filtration residues for re-use. P. A. BOECK, Assr. to CELITE Co. (U.S.P. 1,571,042, 26.1.26. Appl., 19.1.21).—Spent diatomaceous earth filtering material is dried, reduced to fragmentary condition, and heated for a time sufficient to increase the filtration efficiency without substantial removal of chemically-combined water. H. HOLMES.

Drying plant. P. BARDUCCI (U.S.P. 1,571,226—8, 2.2.26. Appl., [A] 19.1.20, [B] 18.2.22, [C] 20.2.22).—(A) A container for the material to be dried is divided into a number of sections, through which separate air currents are passed intermittently and not all at once. The distributing valves or shutters for the air currents and means for operating them in predetermined sequence are placed outside the containers for the material. (B) A drying compartment is provided in the centre with division walls which are H-shaped in plan and do not extend right across the chamber. Air-impelling means are situated opposite one or more openings in the short wall or crossbar of the H, so that air is circulated through the central space and divides right and left through the material in the side spaces, the streams re-uniting to pass through the central space again. (C) In a drying room, reciprocating means are provided for producing vertical air currents, and other means, at the ends of the room, direct air currents respectively towards and from the reciprocator.

B. M. VENABLES.

Fractionating tower. F. B. SAMUEL (U.S.P. 1,571,805, 2.2.26. Appl., 17.4.24).—Vapour is passed downwards through a number of approximately vertical tubes or flues in a heat-insulated vertical casing, to a separator for vapour and condensate at the bottom. The temperature of the tubes is regulated by an upward draught of air around them, the temperature drop within the casing being maintained constant irrespective of weather conditions. B. M. VENABLES.

Decolorising and purifying solutions. INTERNATIONAL SUGAR AND ALCOHOL Co., LTD. (F.P. 596,919, 20.4.25. Conv., 26.6.24).—Lignin obtained by treating cellulosic materials with acids is washed and used in the same way as wood charcoal for purifying liquids. It should not be dried, but if this has occurred, it can be re-activated by steaming. E. S. KREIS.

Carrying out and controlling reactions. E. MÜLLER, and CHEM. FABR. BUCKAU (G.P. 422,159, 1.7.24).—Instead of removing successive samples for analysis in a continuous process, or working a discontinuous process with measured quantities of reagents, the potential difference between a normal electrode and an electrode immersed in the reaction liquid is measured and compared with a standard table. In an example, the application of the process to the preparation of bleaching solutions from chlorine and caustic soda solution is described (cf. B., 1925, 628). E. S. KREIS.

Preparation of stable, dispersible, dry material. E. TRUTZER (G.P. 422,803, 10.9.20).—The material, mixed with a dispersion medium, is ground in a colloid mill until a disperse system of colloidal character is obtained. The medium is then removed by evaporation in an atomising and drying apparatus, and the material recovered as a dust-free dry powder. The process can be used for the preparation of dry colloidal coal.

A. B. MANNING.

Rotary dryer. W. A. HARTY and F. W. MOORE (Re-issue 16,265, 16.2.26, of U.S.P. 1,531,438, 31.3.25. Appl., 16.10.25).—See B., 1925, 577.

Obtaining powders of great fineness. E. PODSZUS (U.S.P. 1,573,017, 16.2.26. Appl., 1.7.22).—See E.P. 183,134; B., 1923, 385 A.

Separation of gaseous mixtures by liquefaction and rectification. E. JORDAN, Assr. to SOC. L'AIR LIQUIDE (U.S.P. 1,574,425, 23.2.26. Appl., 24.4.23).—See E.P. 197,302; B., 1923, 961 A.

Filter. Filtering apparatus. H. S. COE, Assr. to CYCLE Co. (U.S.P. 1,574,556–7, 23.2.26. Appl., 13.8.19 and 26.1.20).—See E.P. 241,453; B., 1925, 975.

Powdered fuel [boiler] furnaces. C. HOLD (E.P. 247,858, 19.9.25).

Electrical gas purification (G.P. 422,619).—See XI.

Filter (U.S.P. 1,568,001).—See XIX.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Effect of sulphur in the briquetting of sub-bituminous coal. H. K. BENSON, J. N. BORGLIN, and R. K. ROURKE (Ind. Eng. Chem., 1926, **18**, 116—117).—The carbonisation for 8 hr. at 700—950° of a mixture in the form of small briquettes containing 3% of crude sulphur, 8% of California asphalt, and 89% of a sub-bituminous coal from Tono, Washington, which was ground to pass 20-mesh, yielded a firm, coke-like mass which, when burned with a forced draught, retained its shape until entirely consumed. The sulphur content of the carbonised residue was found to be about 2·25% after cooling in the retort and about 1·40% after quenching in water. The briquetted mixture also retained its shape while

burning without previous carbonisation. Mixtures of coal and sulphur without the asphalt binder did not coke on carbonisation and mixtures of coal and binder without sulphur produced a friable coke which fell to pieces on burning.

A. W. HOTHERSALL.

Decomposition of humic acids at 100°. W. ELLER and A. SCHÖPPACH (Brennstoff-Chem., 1926, 7, 17—20).—Humic acids decompose with evolution of carbon dioxide and water at temperatures above 80° (cf. A., 1923, i, 542). The amount of decomposition occurring at 100° has been determined for three humic acids, one extracted from a humic coal, and two artificial humic acids prepared respectively from quinol and sucrose. During preparation the temperature of the material was not allowed to rise above 80°, and the humic acids were dried first in air at 80° and then *in vacuo* at 66° to constant weight. This final drying required about 28 days. Between 1 and 2 g. of the dry material was kept at 100° in a slow current of air, the carbon dioxide and water evolved being collected and weighed at 4-hr. intervals over a total period of 52 hrs. Progressive decomposition of all three humic acids occurred, resulting in the evolution of 6—11% of water and 0·6—1·7% of carbon dioxide in 52 hrs., calculated on the original weight of dry material.

A. B. MANNING.

Examination and evaluation of coking coals. R. KATTWINKEL (Gas- u. Wasserf., 1926, 69, 145—150).—The shortcomings of existing methods of determining the caking index of coal are indicated. A method of obviating them consists in subjecting a coke-button prepared from a mixture of coal and sand to a crushing force which is regularly increased by allowing lead shot to fall at a standard rate into a vessel supported on a piston resting on the coke-button. The flow of lead shot is interrupted when the button is fractured, and the amount of shot used is a measure of the caking power of the coal, due allowance being made for uncoked portions of the crucible charge. The results are interpreted in terms of Fischer's theory of coking (cf. B., 1925, 233), which, however, cannot be reconciled with coke-oven results. The influence of size of both sand and coal on caking power are discussed and methods of testing coke are described briefly.

R. A. A. TAYLOR.

Coke production. E. W. SMITH, T. C. FINLAYSON, H. M. SPIERS, and F. S. TOWNEND (Gas J., Coke Number, Nov. 9, 1925, 3—16).—Only for combustion in open grates is the ignitability of coke of any importance; consequently for most purposes (1) suitable grinding and grading, (2) low moisture content, (3) low ash and sulphur contents, and (4) satisfactory combustibility and reactivity, satisfy the requirements. The terms combustibility and reactivity should be restricted to mean respectively combustion with oxygen and reaction with substances other than oxygen. The rate at which coke is consumed in a stove of given size under a constant air blast and starting with a fixed initial temperature is a measure of the combined combustibility and reactivity. Generally this should be high, but for foundry work, where high combustibility and low reactivity are desired, an independent test for reactivity, such as the determination of the rate of reduction of carbon dioxide by the coke maintained at 900°, is necessary. The most important factors influencing the combustibility and the reactivity of coke appear to be its thermal treatment during and after coke formation, its physical properties, and its content of volatile matter. The four methods of operating carbonising plant to give the desired coke discussed are: the blending of coke dust with coal; carbonising blends of caking and non-caking coals; grinding, mixing, and compressing coals prior to carbonisation; and carbonising briquetted blends of coals in vertical retorts. With the adoption of these processes it becomes more necessary to clean the coals. The additional expense involved in the application of these processes would be more than counterbalanced by the enhanced value of a better and more uniform coke, the absence of breeze, greater throughput for a given plant, and the possibility of utilising cheaper coals. By further reducing the time of carbonisation a smokeless fuel containing sufficient volatile matter to render it easily ignitable in an open grate could be produced by these processes even at high temperatures. In the methods of testing coke prominence is given to Rose's method of examining coke structure (cf. B., 1925, 834).

S. PEXTON.

Ignition temperature and reactivity of carbonisation products. K. BUNTE (Z. angew. Chem., 1926, 39, 132—138; Gas- u. Wasserf., 1926, 69, 192—195, 217—218).—The close interdependence of the ignition temperature and reactivity towards carbon dioxide of a variety of carbonisation products (wood charcoal, semi-coke, gas coke, oven coke, coke breeze, graphite), in sizes from 0·5 mm. to 5 mm., is emphasised and illustrated graphically. The ignition temperature is determined by passing a regulated current of air through a column of the material, the temperature of which is gradually raised by means of an electric furnace, and is taken as the temperature at which a thermocouple placed just above the top of the column indicates a sudden rise in temperature. The ignition temperature is raised by increasing the size of the particles of material, and lowered by increasing the air velocity or the oxygen content of the air. It also varies inversely as the adsorptive capacity of the material, *i.e.*, as the surface area exposed per unit mass. The reactivity is measured by passing carbon dioxide over the heated material and determining the degree of reduction to carbon monoxide at different temperatures. By plotting as ordinates the temperatures at which, for the various materials in question, reduction to carbon monoxide has proceeded to an equal degree, against ignition temperatures as abscissæ, an approximately linear relation is found to hold, which enables the reactivity curve of a material to be deduced from a knowledge of its ignition temperature.

W. T. K. BRAUNHOLTZ.

Gaseous combustion at medium pressures. I. Carbon monoxide–air explosions in a closed vessel. II. Methane–air explosions in a closed vessel. R. W. Fenning (Phil. Trans., 1926, A, 225, 331—356).—Pressure–time records have been obtained for the explosion of a series of " complete combustion " mixtures of hydrogen, carbon monoxide, and air, from 100% H_2–air to 50% H_2–air+50% CO–air, in a closed vessel at an initial pressure of 76·8 lb./sq. in., and an initial temperature of 50°. The water vapour content of the gas mixtures was less than 1 in 2000. A second series of records was made to show the effect of addition of water vapour, from 0·3 to 2·56 vols. per 100 vols. of gas mixture, on the explosion of CO–air mixtures. In a third series the effect of a constant water vapour addition (1·2% by vol.) to the first series was observed. The apparatus consisted of an electrically heated mild-steel vessel (7 in. diam. by 8 in. long) with a spark gap ($\frac{1}{2}$ mm.) at the centre, fitted with a diaphragm manometer and pressure-recording device. In the first series just over 2% of H_2–air in the mixture reduced the " explosion time " (time from passage of spark to production of maximum pressure) from 0·26 sec. to 0·1 sec., and 4% reduced it to 0·076 sec. The explosion time for the CO–air mixture itself varied considerably in different determinations (0·246 to 0·29 sec.), due probably to slight variations in humidity. All mixtures gave practically the same maximum explosion pressure. The interval during which a constant maximum pressure is maintained is of the order 0·004 sec. for CO–air mixtures, and varies from 0·001 sec. to zero as the percentage of H_2–air increases. Addition of water vapour has a marked influence on the explosion time of CO–air mixtures, 0·3% by vol. reducing it from 0·29 to 0·16 sec. The same addition of water vapour raises the maximum explosion pressure by more than 2%, whilst further addition up to 2·56% adds only another 1%. Almost identical records were produced by addition of 1·21% of water vapour and of 2·1% of H_2–air respectively to the CO–air mixture. Addition of water vapour in the third series reduces the explosion time when the mixture contains less than 8% of H_2–air, but slightly increases it for greater percentages. Addition of water vapour to all mixtures in which H_2–air exceeds about 8% reduces the maximum pressure by slightly under 1%. Pressure–time records of explosions in methane–air mixtures at an initial pressure of 95 lb./sq. in. and an initial temperature of 100° show that with increase in the methane–air ratio the maximum pressure passes through a maximum at 9·7—10·5% of methane, and the " explosion time " passes through a minimum at 9·7% of methane. Complete records were made for a 9·9% mixture at initial temperatures from 24° to 400°, and at initial charge densities over a range corresponding to pressures from 2 to 5 atm. at 15°. The explosion time is increased by (1) increasing the charge density, (2) lowering the initial temperature, (3) adding a diluent such as water vapour or combustion products. The rise in pressure increases linearly with initial pressure for constant initial temperature. The effect of the variation of specific heat with temperature is apparently masked by other factors.

A. B. Manning.

Desulphurisation of gas. A. Thau (Gas- u. Wasserf., 1926, 69, 125—128).—The history and statistics of dry purification of gas are reviewed and particulars of American practice for removal of hydrogen sulphide by washing with sodium carbonate solution are given. The disadvantage of this wet purification process is its incompleteness and an improved dry process due to Raffloer is described. Dry iron oxide in granular form is fed from hoppers over louvres through which gas leaving the purification system passes. The oxide then falls to the bottom of an incline, whence it is blown by a stream of by-passed gas up a tower down which the main gas stream passes on its way to the vessel via which the oxide enters the plant. The shower of oxide falls down the tower and collects on an inclined false bottom from which it is constantly withdrawn through a self-sealing valve and returned to the feed. Practically no trace of hydrogen sulphide leaves the plant, and the cost per year of treating 50,000 cub. m. of gas per day is M. 30,000, as against M. 275,000 by the ordinary static process. One cub. m. of " Lux " purifying material is capable of purifying 50,000 cub. m. of gas and can be worked up to 70% of sulphur in the fouled oxide.

S. Pexton.

Holzheim shale and shale oil. K. Neubronner (Petroleum, 1926, 22, 57—61).—The oil shale is found in two layers of about 1 m. and 3 m. thickness, respectively. It quickly weathers and crumbles when exposed to the air. The upper layer yields 6—7% of oil, the under layer 1—3%. The mineral is a blue-grey to blackish-blue clayey marl, *d* 2·2—2·3, contains 1—3% of pyrites, and has a content of 16·1% of organic matter in the lower layer. The residue is basic, contains 15—17% CaO, and is used in the manufacture of cement. The shale after crushing is distilled in an internally-heated retort in the presence of inert gas. 200 Cals. are required for the distillation of 1 kg. of shale, and 70—80% of the oil is recovered. The oil, *d* 0·960—0·965, is dark brown, of an unpleasant odour due to sulphur compounds, and has a large content of unsaturated hydrocarbons. The content of aromatic hydrocarbons is small, of basic substances 0·5%, of phenols 1·4%, and of paraffin wax 0·7—0·8%; 66% distils over up to 360°. The elementary analysis shows carbon 82·6% and hydrogen 10·1%. H. Moore.

Chemical composition of Rumanian petroleum oils. N. Danaila (Petroleum, 1926, 22, 209—210).—The determination of unsaturated hydrocarbons was carried out by the direct method by means of ozone (cf. B., 1924, 857). The aromatic hydrocarbons were determined by the use of sulphuric acid of concentration 98·33% H_2SO_4. In the fraction 65—150° (11·31%), *d* 0·7542, there were found 1·46%, 20·42%, and 78·12%; in the fraction 65—95° (3·41%), *d* 0·7294, 1·65%, 17·50%, and 80·85%; in the fraction 95—125° (4·99%), *d* 0·7594,

1·39%, 20·92%, and 77·69%; in the fraction 125—150° (2·91%), d 0·7728, 1·22%, 23·98%, and 74·80%; and in the fraction 150—300°, 1·64%, 16·89%, and 81·47% respectively of unsaturated, aromatic, and saturated hydrocarbons. H. Moore.

Action of petroleum-refining agents on naphtha solutions of pure organic sulphur compounds. A. E. Wood, C. Sheely, and A. W. Trusty (Ind. Eng. Chem., 1926, 18, 169—171).—The refining agents used were sulphuric acid (fuming, d 1·84, and d 1·58), sodium plumbite, sodium hydroxide, silica gel, fuller's earth, and alumina. Stock solutions of sulphur derivatives (*iso*amyl mercaptan, hydrogen sulphide, methyl sulphate, methyl *p*-toluenesulphonate, carbon disulphide, *n*-butyl sulphide, *n*-propyl disulphide, thiophen, diphenylsulphoxide, *n*-butyl sulphone, and free sulphur) were made in a sulphur-free petroleum distillate, d^{20} 0·77. The general procedure was to treat 50 c.c. of the stock solution with two quantities of the reagent (4 and 16 c.c. respectively) for a period of 1 hour, the desulphurising efficiency being measured by the decrease in sulphur content after treatment. Under these conditions sulphuric acid of all strengths was quite ineffective towards free sulphur or carbon disulphide, but the other sulphur compounds were removed fairly easily except in the case of the weaker acid. Sodium plumbite removed hydrogen sulphide and alkyl sulphate, and to a certain extent the mercaptans as lead mercaptides. Sodium hydroxide behaved similarly to sodium plumbite. Silica gel was effective in most cases, except for free sulphur, hydrogen sulphide, carbon disulphide, and thiophen. Fuller's earth and alumina behaved similarly and were less effective than silica gel. The action of the last three agents is due to adsorption only. The effects of each agent on each sulphur compound are tabulated. S. Bowman.

Adsorption studies on decolorisation of mineral oils. T. H. Rogers, F. V. Grimm, and N. E. Lemmon (Ind. Eng. Chem., 1926, 18, 164—169).—Working with three widely different petroleum products—kerosene, wax, and naphtha solution of cylinder stock—the authors have studied the behaviour of various adsorbents such as clays, activated charcoal, and inorganic gels. The oil was agitated with the adsorbent in definite proportions at room temperature for definite periods, and after filtration colour determinations were made. For colour measurement and expression of results the true colour method of Parsons and Wilson (B., 1922, 402 A), using the Duboscq instrument, was adopted. Applying Freundlich's equation, $x/m=KC^{1/n}$, where x is the amount adsorbed by m g. of adsorbent, C the equilibrium concentration, and n and K are characteristic constants, the logarithmic values of colour units removed were plotted against equilibrium colour and gave a series of characteristic curves for each product. The use of this equation affords a convenient method of evaluating the decolorising properties of adsorbents. The colour formation in cracked distillates when treated with certain adsorbents (Dunstan, Thole, and Remfry, J.S.C.I., 1924, 179 T) is due partly to polymerisation and partly to oxidation. S. Bowman.

High-vacuum distillation of mineral oils. L. Steinschneider (J. Inst. Petrol. Tech., 1925, 11, 514—523).—The distillation of petroleum is not accompanied by serious decomposition till the temperature exceeds 300—320°. Above this temperature decomposition rapidly increases, and affects the quality of the lubricating oil fractions. The boiling temperature may be lowered by the use of steam or by diminished pressure. The optimum amount of steam is 1·65 pts. per 1 pt. of oil distillate, but in practice about 3·5 pts. are used. Higher temperatures prevail in bottom-heated stills than in those with internal flue, in consequence of the height of the column of oil above the heating surface. Plant for vacuum distillation at absolute pressures as low as 5 to 25 mm. operates with satisfactory results. The author advocates high vacuum, minimal use of steam, active circulation, the avoidance of re-distillation and, as far as possible, of chemical treatment of the distillates. Vacuum distillation is particularly applicable to inferior heavy asphaltic oils. It effects an economy in fuel of 50%, an extra yield of 4% in distilling and 4—6% in refining, and a saving of 50—75% in sulphuric acid. H. Moore.

Refining of mineral oils. H. I. Waterman (J. Inst. Petrol. Tech., 1925, 11, 576—582).—The presence of sulphur or halogens in oils leads to the separation of dark substances containing carbon. Unsaturated substances are not, however, as harmful in this respect as was once thought. Free or combined sulphur causes kerosene to burn with a smoky flame, as also do halogen compounds, such as ethyl bromide. Kerosene heated to 240° for two days in a Carius tube with ethyl sulphide became deep brown in colour. Kerosene was attacked by chlorine at room temperature with evolution of hydrogen chloride, and after heating had the consistency of asphalt and contained free carbon. Paraffin wax of m.p. 50·6° was cracked under pressures varying from 7 to 219 atm. The content of unsaturated compounds in the product was greater in proportion as the pressure was lower; least at the highest pressure in the presence of hydrogen. Extraction with liquid sulphur dioxide divides the compounds present in petroleum into two groups, which may be subjected to separate refining. In a petroleum fraction of d 0·86—0·92, treatment with 54% of silica gel reduced the sulphur content by 20%, whilst the use of 114—117% of gel by weight brought about a reduction of 50—70%. In the use of the hypochlorite process it is important to regulate the quantity of free alkali present. H. Moore.

Edeleanu process [of petroleum refining]. H. I. Waterman and J. N. J. Perquin (J. Inst. Petrol. Tech., 1925, 11, 560—570).—Kerosene distillates, transformer oils, Russian and medicinal oils, were treated by the Edeleanu process with

liquid sulphur dioxide. In the case of kerosenes this treatment was followed by treatment with 0·5% of sulphuric acid and 2% of oleum, with excellent results. Sulphur dioxide has great desulphurising power, the sulphur content of kerosenes being reduced from 0·29% to 0·019%, from 0·32% to 0·01%, and from 1·13% to 0·038% respectively by the above treatment. Silica gel treatment and the hypochlorite process did not remove sulphur and unsaturated constituents to the same extent. Treatment with 2% and 5% of sulphuric acid *d* 1·84, and refining with silica gel does not considerably raise the aniline point (cf. Tizard and Marshall, J.S.C.I., 1921, 20 T). Sulphur dioxide treatment raises it, *e.g.*, from 52° to 65·2°. This treatment gives favourable results as regards colour, and colour after exposure to a quartz mercury vapour lamp. By sulphur dioxide and acid treatment the sludge value of transformer oil was reduced from 1·3% to 0·5%. Sulphur dioxide may also be used in refining vegetable and animal oils and fats. H. MOORE.

Separation of the components of petroleum. Bromination of Persian petroleum fraction, b.p. 60—80°. P. F. GORDON, D. BAIRD, and T. G. HUNTER (J. Roy. Tech. Coll. Glasgow, 1925, [2], 53—63).—When the fraction of Persian petroleum boiling between 60° and 80° is treated with bromine, drop by drop, in the presence of an excess of iron at 16° smooth bromination occurs without any side-reactions. The product is a plastic mass, the bulk of which dissolves in ether leaving a white crystalline residue. After distillation of the ether solution the residual liquid separates into two immiscible layers, and a small amount of a white crystalline substance (m.p. 164°) is precipitated. The heavy liquid has the empirical formula $C_5H_7Br_3$ and the lighter liquid the formula $C_6H_{10}Br_4$. Both liquids are viscous and have a tendency to decompose with evolution of hydrogen bromide. The ether-insoluble crystals can be fractionated from ethylene dichloride into five crystalline products having the following melting points in order of increasing solubility: 293·5°, 273·5°, 299·8°, 283°, and 305°. The second and third have the empirical formulæ $C_7H_6Br_4$ and $C_5H_4Br_4$ respectively. All five compounds are soluble in carbon disulphide and in acetone and are not decomposed by an alcoholic solution of potassium hydroxide. The first bromide on shaking with benzoyl chloride and pouring the product into water containing a little sodium carbonate, gives a white crystalline precipitate, soluble in water, alcohol, and ether. After recrystallisation from alcohol it melts at 120°. A. R. POWELL.

Extension of method of critical temperatures of solution to analysis of petrols. M. AUBERT and E. AUBRÉE (Compt. rend., 1926, **182**, 577—579; cf. Simon and Chavanne, A., 1919, ii, 267, 432, 433).—In the earlier work the temperature above which mixtures of aniline and hydrocarbons yielded homogeneous mixtures no matter what the composition, served as a basis for summary analysis, *i.e*, the evaluation of percentages of aromatic, saturated cyclic and acyclic hydrocarbons present in a given petroleum. The method necessitated the nitration of aromatic substances and re-determination of the critical temperature of solution. It has now been shown that benzyl alcohol may be substituted for aniline and similar data obtained. By means of solving simultaneous equations obtained by using aniline and benzyl alcohol the composition of petrol may be determined without recourse to nitration. R. A. MORTON.

Ignition of carburetted mixtures [motor fuels] by adiabatic compression. A. PIGNOT (Compt. rend., 1926, **182**, 376—377; cf. A., 1907, ii, 18, 946; J.C.S., 1914, 2027—2036).—Mixtures of dry air and *n*-heptane, *n*-hexane, *n*-octane, *cyclo*hexane, benzene, toluene, *m*-xylene, mesitylene, and ethyl alcohol have been subjected to a fixed volumetric compression and the initial temperature necessary for ignition has been determined. By plotting ignition temperature against percentage of hydrocarbon or alcohol, curves are obtained which sharply differentiate between aromatic and saturated hydrocarbons. With a volumetric compression of 9 benzene and absolute alcohol give similar curves. Increasing amounts of water in the alcohol result in a flattening of the curve and the disappearance at 40% of water of the minimum which is observed in this and other cases; *cyclo*hexane–benzene mixtures show no anomalies in the curves. Addition of 10% of toluidine or ethyl sulphide (which are known as very active anti-detonators) to *cyclo*hexane exerted negligible effects on the characteristic curves. R. A. MORTON.

Report of Surgeon General's committee [U.S. Public Health Service] on lead tetraethyl. (Ind. Eng. Chem., 1926, **18**, 193—196).—Some two hundred men were examined to determine whether lead tetraethyl blended with petrol had any effect as regards lead absorption. The average time of exposure to the substance was two years. The methods employed were the determination of the lead content of fæces of workers, the estimation of the number of stippled red cells in the blood of workers, and the measurement of the strength of the extensor muscle of the forearm. Drivers of cars using the blended fuel showed no signs of lead absorption. Garage workers handling the fuel showed very slight signs of lead absorption, but these were extremely small compared with the effects on workers known to be exposed to lead poisoning risks in other industries. The committee concludes that no reason exists for prohibiting the use of lead tetraethyl but proposes to prolong its investigations. W. N. HOYTE.

Potential public health hazards from the use of "ethyl-gasoline." F. B. FLINN (J. Ind. Hyg., 1926, **8**, 51—66).—Absorption of lead tetraethyl occurs when "ethyl-gasoline" is applied to the skin of rabbits, guinea pigs, and goats, and also when animals are exposed to the fumes resulting from the evaporation of the gasoline in a gas chamber; similar results were obtained when the substance was allowed to evaporate in the open air. Exposure to the vapour results in more rapid absorption than does

application to the skin, and the rate of excretion of the lead is less than the rate of absorption.
H. J. Channon.

Portable combustion apparatus for detection and measurement of small quantities of petroleum vapour. C. A. Neusbaum, P. L. De Verter, and E. W. Dean (Ind. Eng. Chem., 1926, **18**, 183—185).—General requirements and known standard apparatus for the measurements are discussed. The authors describe an apparatus depending on the shrinkage in volume of a known volume of air and petroleum vapour after exposure to a red hot platinum wire. The apparatus is calibrated by means of mixtures of known composition. W. N. Hoyte.

Cracking solid paraffins with formation of low-boiling products, using activated carbon. H. Herbst (Z. angew. Chem., 1926, **39**, 194—196).—A solid paraffin (m.p. 50—52°), distilled in contact with activated carbon at about 300° under atmospheric pressure, yielded 22% of a light oil, b.p. 45—150°, 13·4% of a heavier oil, b.p. 150—200°, and 21·2% of oil of b.p. 210—300°, reckoned by weight on the solid raw material. Further quantities of light oil could be obtained by subjecting the oily distillate distilling above 300° and the unchanged paraffin to a further distillation with activated carbon, giving a total yield of 27% of light oils distilling below 150°. This compares very favourably with the yield of 4—12% obtained by cracking crude petroleum. The process can be carried out at atmospheric or reduced pressure for paraffins boiling above 300°, which simplifies the plant required in practice, while no appreciable quantities of coke or asphalt are produced, thereby prolonging the life of the equipment. Materials with a b.p. below the decomposition temperature must be distilled under a pressure (10—25 atm.) sufficient to raise the b.p. to the required temperature, generally about 300°. B. W. Clarke.

Lubricating oil for electricity meters. D. Holde and R. Schachenmeier (Petroleum, 1926, **22**, 161—168).—Types of meters and the characteristics of a satisfactory lubricating oil are discussed. A viscosity of 20° Engler and an iodine value of 6 are suggested as upper limits. Experiments are being continued as regards volatility, resinification, oxygen absorption, and surface tension.
W. N. Hoyte.

Deterimination of the melting points of asphalts. I. J. Lagerqvist and M. Fredman (Svensk Kem. Tidskr., 1925, **37**, 266—276; Chem. Zentr., 1926, 1., 1339—1340).—The Kraemer–Sarnow method of determining the m.p. of asphalts (B., 1903, 291) gives discrepant results with only slight changes in the experimental conditions. The modification due to Barta (B., 1911, 1367) is recommended. A uniform rate of heating of 1° per min. should be used. The time allowed to lapse between filling the tube and making the determination, and cooling of the sample after filling the tube have little influence on the result. The apparent m.p. rises linearly with the depth of the layer of material in the tube. The internal diameter of the tube should not vary more that±0·2 mm. from the standard (6·5 mm.). The quantity of mercury used has considerable influence, and should therefore be accurately measured.
A. B. Manning.

Reactivity of coke. J. P. Arend and J. Wagner (Fuel, 1926, 5, 106—116).—See B., 1924, 973.

See also A., Mar., 246, **Reaction provinces** (Jorissen). 250, **Union of carbon monoxide and oxygen in contact with nickel, copper, and their oxides** (Bone and Andrew). 267, **Detection and determination of gaseous olefines** (Sorokin and Belikov). 286, **Naphthenic acids** (Naphtali).

Expansion of pitches and asphalts. Mallison Jacobsohn, and Sarre.—See III.

Petroleum insecticides. Gray and De Ong.—See XVI.

Patents.

Manufacture of artificial fuel. E. C. Marcesche (E.P. 239,474, 27.11.24. Appl., 8.9.24).—Anthracene or naphthalene oil or paste is atomised by means of a jet of air and steam and projected on to a mixture of fuel and pitch binder, or on to the pitch binder alone, the pulverised fuel being added subsequently. The mixture is briquetted.
R. A. A. Taylor.

Method of treating coal products. W. E. Trent, Assr. to Trent Process Corp. (U.S.P. 1,570,103, 19.1.26. Appl., 23.9.19. Renewed 18.7.25).—Coal, in a very finely divided state, is mixed with a fluxing agent, and fed into a moving fluid current. The mixture is conveyed through a heating zone, in which the volatile by-products are removed from the coal and the ash unites with the fluxing agent. The carbon content of the coal, free from its volatile matter, is then collected.
A. B. Manning.

Dust removal in lignite briquetting works. Siemens-Schuckertwerke G.m.b.H., Assees. of C. Hahn (G.P. 422,148, 16.3.22).—The dry dusty air from the interior of the works is mixed with the moist fumes from the drying apparatus, and the solid particles in the mixture are separated by electrical precipitation. The removal of dust is more complete than by purely mechanical processes, and the risk of explosion is avoided.
A. B. Manning.

Charging coke ovens. Collin und Co., and J. Schaefer (E.P. 227,470, 12.1.25. Conv., 12.1.24). —A dense coke is formed by charging coal into ovens from a greater height than usual, by suitably disposing the hoppers, or by providing a hopper which can be raised. The charge openings form such a large proportion of the cross-section of the ovens that the coal looses none of its momentum in passing through and packs tightly in the ovens. In addition fireclay slabs connected to the under side of the charging doors in such a way that they are free to move vertically, bear down upon the coal charge during coking. S. Pexton.

Retorts [for carbonising]. MERZ AND MCLELLAN, and E. G. WEEKS (E.P. 246,319, 4.2.25).—An apparatus for charging and levelling the fuel in vertical retorts is described. It consists of a rotary frame carrying fixed scrapers in a horizontal plane at the upper level of the fuel. One scraper is plough-shaped and throws part of the fuel over against the retort wall and the remainder inwards to the centre of the retort. The second scraper is separated from the first by an angle approximating to 180° and describes a circle of smaller radius than the plough. An inclined charging shoot which is carried by the frame and into which fuel is fed by a worm, through a gas-tight roller valve, drops coal into the retort in advance of the plough scraper. The fuel height is indicated by a pointer connected by a lever system to a metal flap which is connected to the rotating frame like a flag which trails over the fuel surface. S. PEXTON.

Apparatus for manufacture of water-gas. HUMPHREYS AND GLASGOW, LTD., Assees. of C. S. CHRISMAN (E.P. 245,688, 19.8.25. Conv., 18.4.25).*—An automatically operated gas valve obviating the use of all hot valves in water-gas manufacture, has three gas connexions, one, 20 (Fig. 3), to the wash-box on which it stands and the other two, 13 and 12, to the bottom of the generator and waste-

FIG. 1.

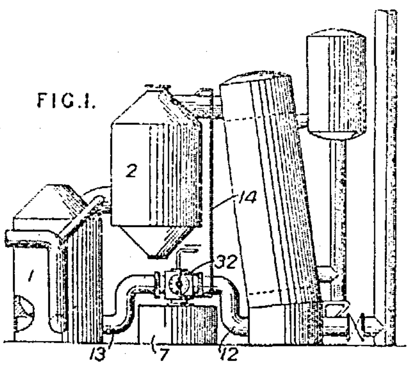
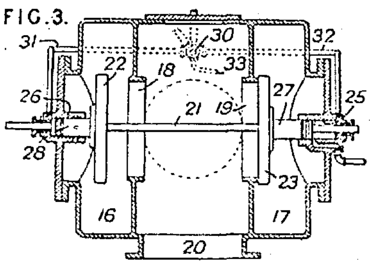


FIG. 3.



heat boiler outlet respectively. The direction of gas flow is governed by the position of a common spindle, 21, carrying two disc valves, 22 and 23. The spindle is moved by the steam pressure acting on one or other of the two pistons, 26 and 27. A hand-operated four-way steam cock, 30 (Fig. 3), controls the supply of steam to the plant. When the cock, 30, is in the left-hand position shown in Fig. 3, steam travels along 32, 25, and 14, and through the hot chequered chamber to the top of the water-gas generator and down through the latter. Valve, 23, remains seated because of the differential pressure between the top and bottom of the generator, and the water-gas made enters the wash-box via 13, 18, and 20. When the cock, 30, is in the right-hand position steam passes along 31, moves the piston and valve spindle to the right until when valve, 22, is closed, steam enters 16 and passes via 13 to the bottom of the generator. Valve, 22, remains closed because of the differential pressure across the fuel bed, and the water-gas made having passed the chequered chamber and waste-heat boiler enters the wash box via 12 and 20. S. PEXTON.

Apparatus for purifying gas. H. T. GRAFTON, C. W. WEESNER, and F. J. HOFFMAN (U.S.P. 1,572,599, 9.2.26. Appl., 30.3.21).—The gas is passed into a preheater arranged in a furnace so as to be heated by the furnace gases, and is delivered therefrom to purification chambers suspended from the furnace roof. T. H. BURNHAM.

Purification of coke-oven gas. SOC. ANON. DES ANC. ÉTABL. EGROT ET GRANGÉ (F.P. 593,565, 28.4.24).—The gases are led up through a vertical scrubber down which passes cold ammonia liquor containing tar. The gases extract the ammonia and tar, and pass then through a system of condensers where the tar, water, and part of the ammonia are separated. The liquor separating in the condensers is treated with lime and returned to the scrubber. A. B. MANNING.

Separation of condensable products [from distillation gases] by electrical precipitation. SIEMENS-SCHUCKERTWERKE G.M.B.H. (G.P. 422,121, 13.12.21).—Distillation gases are led in succession through a series of electrical precipitation chambers, which are maintained at temperatures corresponding to the dew point or condensation temperature of the products to be separated therein. Before entry into each chamber, the gas is mixed with some of the product previously condensed therein. This forms a simple means of maintaining the required temperatures in the chambers. A. B. MANNING.

Gas purifying agent capable of regeneration. SIRIUS WERKE A.-G., and J. BRUNNER (G.P. 422,630, 14.3.25).—Pipe-clay impregnated with oil is used as a purifying agent for gas, in particular for the removal of naphthalene. A. B. MANNING.

Purifying agent for acetylene. CHEM. FABR. GRIESHEIM-ELEKTRON (F.P. 596,840, 16.4.25).—Basic calcium or magnesium hypochlorite, mixed with water and a binding material, is used as a purifying agent for acetylene. A. B. MANNING.

Ascertaining the proportion of a constituent in a fluid. [Determining carbon dioxide in flue-gas.] E. X. SCHMIDT and U. O. HUTTON, Assrs. to CUTLER-HAMMER MANUF. Co. (U.S.P. 1,569,682, 12.1.26. Appl., 30.1.24).—The ends of a conducting wire, the middle portion of which is

coiled over a roller, pass down centrally through two vertical tubes placed side by side and are maintained in a state of tension by springs at the lower ends of the tubes. Air is passed up one tube and the flue-gas under examination up the other, both gases being first brought to the same temperature, and either dried, or saturated with water vapour. The wire is heated by passing an electric current through it. Owing to the relatively low heat conductivity of carbon dioxide, the portion of the wire in the tube through which the flue-gas is passing becomes hotter than the portion in the other tube and expands, in consequence, to a greater extent, so that its tension becomes less than that of the other portion. The roller turns to equalise the tensions, and by affixing an indicator to the roller and noting its movements a measure can be obtained of the percentage of carbon dioxide in the flue-gas. T. S. Wheeler.

Motor fuel. Farbw. vorm. Meister, Lucius, u. Brüning, Assees. of P. Roth (G.P. 421,814, 18.12.23).—The acetals of aldehydes and alcohols, in particular those of acetaldehyde and formaldehyde, or of ethyl and methyl alcohols, are used as motor fuels, either alone or mixed with other organic liquid fuels. They form a substitute for benzine or benzene of about equal value. A. B. Manning.

Cracking hydrocarbon oils. H. Wolf (E.P. 215,336, 16.4.24. Conv., 1.5.23).—Heavy hydrocarbon oil is passed at a pressure of 20—300 atm. through a steel coil in which it is subjected to a temperature of 400—450° for 30—80 secs. The cracked vapours are expanded through a valve into a fractional condensing apparatus, in which the oil to be treated is preheated directly or indirectly. Any high-boiling oil separated is returned to the cracking apparatus. The apparatus is rapid and safe in action, and there is little production of coke and gas. In one experiment 115 litres of oil per hr. were passed at a linear speed of 2·5 m. per sec. under a pressure of 300 atm. through a coil in which the oil was heated to 430° for 50 sec., when 33 litres of benzine boiling up to 150° were obtained. Linear oil speeds of at least 1 m. per sec. are necessary to prevent deposits in the coil. T. S. Wheeler.

Cracking hydrocarbons. F. B. Koontz, Assr. to Cosden and Co. (U.S.P. 1,567,062, 29.12.25. Appl., 8.5.24).—Petroleum oil is passed at a pressure of about 200 lb./in.^{2} through a series of pipes heated to 470°, and the cracked vapours are led directly into a long cylinder in which carbonaceous matter and high-boiling oil are separated. The uncondensed vapours pass up a long vertical pipe, where any entrained carbon separates and falls back into the cylinder, and thence into a reflux condenser. The vapours coming from the top are cooled by cold oil to be treated, the condensing action being controlled so that about 75% of the vapours passing to the condenser are liquefied and withdrawn from the bottom. While in the condenser they are mixed with 33% of their weight of fresh preheated oil from the top cooler. The mixture is passed back to the cracking apparatus. The vapours from the cooler on top of the reflux condenser are condensed and recovered. The continuous removal of carbonaceous matter in the cylinder, whence it is discharged at intervals, enables the apparatus to be run continuously for long periods without cleaning. T. S. Wheeler.

Distilling and blending hydrocarbons. D. L. Newton (U.S.P. 1,567,457–8, 29.12.25. Appl., 19.7.24).—(a) An absorbing medium containing hydrocarbons from casing-head gas is treated for the production of a stabilised motor fuel by partial distillation, followed by repeated counter-current contact between the liquid medium and a mixture of the distilled vapours with steam. (b) An apparatus for the above purpose comprises a tower with a distilling chamber at the top and a succession of combined distilling and blending chambers below, each of the latter being provided with perforated bottoms having perforated re-circulation tubes over the perforations. From the distilling chamber at the top the liquid runs downwards step by step through the tower. The vapour is mixed with steam and conducted to the bottom of the tower, up which it passes step by step, being exhausted just below the distilling chamber. B. M. Venables.

Oil [cracking] still. J. Primrose, Assr. to Power Specialty Co. (U.S.P. 1,568,182, 5.1.26. Appl., 2.7.21).—Oil is cracked by passing it through a pipe coil heated in a cracking chamber which is partially separated from the combustion chamber of the furnace by a bridge wall. The oil after passing through the coil in the cracking chamber passes through a series of pipes placed just inside the roof of the cracking chamber and then through a series of pipes built into the roof of the combustion chamber. The temperature of the roof is thus reduced and overheating of the cracking coil with consequent carbonisation of the oil, due to radiation of heat from the roof, is minimised. T. S. Wheeler.

Refining mineral oils. H. Stinnes-Riebeck Montan- u. Oelwerke A.-G. (G.P. 421,858, 4.12.24).—Mineral oils are refined by the action of tin tetrahalides in a finely divided or diluted form. The tin tetrahalide may be used precipitated on a finely divided material. After the reaction a basic material is added, which serves to remove the acid formed and to bleach the oil. By this process too rapid a reaction and the consequent formation of gums is avoided. A. B. Manning.

Fuel briquettes. J. F. O'Donnell (E.P. 247,272, 10.11.24).—See U.S.P. 1,557,320; B., 1925, 949. The temperature specified is 800—1200°.

Process of manufacturing fuel. W. E. Trent (E.P. 228,862, 17.11.24. Appl., 9.2.24).—See U.S.P. 1,512,427; B., 1925, 62.

Pulverising, feeding, and burning fuel. J. Crites, Assr. to Raymond Bros. Impact Pulverizer Co. (U.S.P. 1,573,040, 16.2.26. Appl., 8.3.24).—See E.P. 230,434; B., 1925, 870.

Apparatus for distilling carbonaceous materials. C. A. Griffiths (U.S.P. 1,573,824, 23.2.26. Appl., 31.5.24).—See E.P. 228,749; B., 1925, 275.

Gas producer. C. W. Lummis and G. H. Isley, Assrs. to Morgan Construction Co. (Reissue 16,262, 16.2.26, of U.S.P. 1,251,064, 25.12.17. Appl., 13.7.23). —See B., 1918, 117 A.

Method and means for using motor fuels. T. Midgley, jun., Assr. to Gen. Motors Corp. (U.S.P. 1,573,846, 23.2.26. Appl., 15.4.22).—See E.P. 196,237; B., 1924, 165.

Distilling volatile products [mineral oils]. L. Granger, C. Mariller, and C. Prache (U.S.P. 1,574,622, 23.2.26. Appl., 9.1.20).—See E.P. 154,558; B., 1922, 4 A.

Washing minerals or coal. G. Ranwez (E.P. 237,928, 4.8.25. Conv., 2.8.24).

Charging of coal into coke-ovens. Collin und Co., and J. Schaefer (E.P. 234,053, 21.1.25. Conv., 15.5.24. Addn. to 227,470).

See also pages 256, **Dispersible dry material** (G.P. 422,803). 264, **Purification of hydrocarbons** (E.P. 246,210); **Purification of oils** (G.P. 421,909). 276, **Bituminous compositions** (E.P. 246,548); **Bituminous emulsions** (U.S.P. 1,567,061). 284, **Electrode carbon** (G.P. 422,235). 299, **Lead tetraethyl** (U.S.P. 1,567,159).

III.—TAR AND TAR PRODUCTS.

Determination of the coefficient of cubical expansion of pitches and asphalts. H. Mallison, F. Jacobsohn, and K. Sarre (Z. angew. Chem., 1926, **39**, 154).—A flask of about 200 c.c. capacity, carrying a ground-in stopper of elongated, tubular shape, is filled to a mark on the stopper with pure, air-free water at 15° and weighed. It is then emptied, dried, and about 100 g. of the sample are accurately weighed into it, water again being added to make up to the mark. This is done at 15° and 60°, and the flask and its contents are weighed on each occasion. The coefficients of cubical expansion for samples of coal-tar pitch and petroleum asphalt, between 15° and 60°, were found to be 0·00045 and 0·00062 respectively. W. T. K. Braunholtz.

Patents.

Treatment of low-temperature tar from lignite. Soc. Anon. Hydrocarbures et Dérivés (F.P. 596,143, 3.7.24).—The dry tar is treated with a solvent, such as 90% alcohol, which dissolves the phenols and part of the light hydrocarbons. The mixture is filtered from the residue which contains paraffin, and the filtrate is fractionated to separate the solvent, phenols, and light oils. The crude phenols may be fractionated to give phenol, cresols, xylenols, etc., or may be hydrogenated to *cyclo*hexanols. The light oils are cracked or hydrogenated in the presence of a desulphurising agent, giving motor fuels similar to benzol. The residue is separated into a paraffin and non-paraffin fraction by treatment with acetone. The crude solid paraffins are pressed and purified by treatment with superheated steam. From the second fraction lubricating and fuel oils are obtained by distillation in vacuum. A. B. Manning.

Purification of benzol and similar hydrocarbons. K. Cox and P. J. McDermott (E.P. 246,210, 21.10.24).—The refining of benzol, petrol, and other light distillates is carried out by distillation in the presence of an adsorbent material such as bauxite, kaolin, soapstone, and similar micro-porous materials. The adsorbent material, ground to pass a 200-mesh sieve, is placed in the still without any previous treatment, agitation being effected by ebullition or by mechanical means. This combination of distillation and refining is claimed to eliminate the gum-forming constituents without removing the low-boiling unsaturated hydrocarbons. The spent adsorbent earth can be reactivated by roasting or acid treatment. S. Bowman.

Purification of oils. F. Hofmann and M. Dunkel (G.P. 421,909, 12.12.23).—Mineral and tar oils and low-temperature tars are refined by heating under pressures of less than 100 atm., or without pressure, in the presence of catalysts, or in vessels with catalytically active walls. The products are stable in air and light, and in most cases do not require further treatment with sulphuric acid. A. B. Manning.

Recovery of acetone from light oils. Zeche M. Stinnes, Assees. of A. Weindel and H. Kiemstedt (G.P. 421,910, 7.2.24).—Benzol first washings are washed with a quantity of water which is used repeatedly until it has dissolved a suitable quantity of acetone, when the acetone is expelled by steam. The distillate is treated with sodium bisulphite solution, whereby evil-smelling impurities are driven off by the heat of reaction, and the acetone–bisulphite compound is worked up in the usual manner. L. A. Coles.

Continuous distillation of tars and oils. E. Blümner (U.S.P. 1,573,370, 16.2.26. Appl., 11.4.21). —See G.P. 340,991; B., 1922, 407 A.

IV.—DYESTUFFS AND INTERMEDIATES.

Red and green shades of Indanthrene Blue RS and GCD. A. Zimmerfeldt (Chemicals, 1925, **24**, 19—20; Chem. Abstr., 1926, **20**, 293.— Experiments are given supporting the view that the redder shade of Indanthrene RS and GCD is caused by the oxidation of the carboxylic hydrogen atom, whilst the greener shade is due to that of the imino-hydrogen atom of the hydrazine group. A. A. Eldridge.

Effect of dyes used in paper-making on the animal life of water. O. Haempel (Z. Unters. Nahr. Genussm., 1925, **50**, 423—426).—Experiments

have been carried out to determine the toxicity to the animal life of water of eleven solutions of dyes used in paper-making. Dilutions of 1 part of dye to 100,000, 500,000, and 1,000,000 parts of water, respectively, were taken and their effects noted daily in each case for 10 days. The dyes have been placed in four classes according to their toxicities, Orange II. and Safranine being harmless, whilst Victoria Blue, Brilliant Green, and Benzol Green are the most poisonous investigated. J. GRANT.

Four cases of poisoning by chloronitrobenzene and one by acetanilide, occurring in a chemical works. A. RENSHAW and G. V. ASHCROFT (J. Ind. Hyg., 1926, 8, 67—73).—Exposure to trays into which a mixture of *o*- and *p*-chloronitrobenzene was run at 90° and allowed to cool, caused poisoning usually within three days, apparently due to absorption of vapour through the lungs; similar symptoms were observed in a case of acetanilide poisoning, and it is suggested that aniline may be the toxic substance (produced in the former case by the reducing action of the hæmoglobin of the blood), and that the formation of a stable compound with hæmoglobin impairs the oxygen-carrying capacity of the blood. H. J. CHANNON.

PATENTS.

Manufacture of new [azo] dyestuffs. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (E.P. 246,394, 7.8.25).—Mordant, acid, and direct cotton dyestuffs are obtained from β-naphthylamine-4 : 5-dicarboxylic acid (known in the free state only in the form of its anhydride), either by coupling it with a diazo compound or by diazotising it and coupling it with a suitable component. The dyestuffs may be converted into yellow, orange, and violet acid or/and mordant dyestuffs containing chromium by treatment with chromium compounds, and green mordant dyestuffs of particular importance are similarly obtained from those dyestuffs produced by coupling β-naphthylamine-4 : 5-dicarboxylic acid with *o*-hydroxydiazo-compounds. For example, an acid *dyestuff* which yields brick-red shades on wool very fast to washing, and also to potting when after-chromed, is obtained by coupling (after diazotisation) 8·5 pts. of aniline-4-sulphonic acid with 10·7 pts. of β-naphthylamine-4 : 5-dicarboxylic anhydride dissolved in 150 pts. of water containing 10 pts. of 30% caustic soda and 10 pts. of soda ash. A green *dyestuff* suitable for printing on cotton mordanted with chromium or for dyeing fast green shades on wool from an acid bath is prepared from the azo dyestuff obtained from diazotised 4-nitro-2-aminophenol-6-sulphonic acid and β-naphthylamine-4 : 5-dicarboxylic acid, by boiling 47·6 pts. with 950 pts. of water and chromium fluoride (equiv. to 22·6 pts. of Cr_2O_3), the product being purified by solution in dilute caustic soda, and precipitation by subsequent addition of acetic acid. A. J. HALL.

Vat dyes of the thioindigo series. A. W. JOYCE, Assr. to CHEMICAL FOUNDATION, INC. (U.S.P. 1,568,458, 5.1.26. Appl., 28.1.25).—6-Chloro-2 : 3-diketo-4-methyldihydro-1-thionaphthen or one of its 2-keto-equivalents is condensed, for example, by heating in a solvent such as glacial acetic acid, benzene, nitrobenzene, or the like, with or without addition of a condensing agent such as zinc chloride or acetic anhydride, with a 3-oxy-1-thionaphthen compound, *e.g.*, its alkoxy- or alkoxy-halogen derivatives, to yield dyestuffs which give on animal and vegetable fibres, in an alkaline hyposulphite vat orange-red to bluish-red shades fast to light, chlorine, acids, and alkalis. These dyestuffs can be further halogenated to give bluer shades. For example, 6-chloro-3-keto-4-methyldihydro-1-thionaphthen-2-oxime is heated in glacial acetic acid at 100° with 4-bromo-3-oxy-6-ethoxy-1-thionaphthen to give a product dyeing soaped cotton in a vat bluish-red shades. For halogenation the dyestuffs are dissolved in sulphuric acid and treated directly with chlorine or bromine. T. S. WHEELER.

Carbocyanine dyes [infra-red sensitisers]. G. O. GUTENKUNST, Assr. to EASTMAN KODAK CO. (U.S.P. 1,568,667, 5.1.26. Appl., 29.9.21).—A β-naphthaquinaldine alkyl halide is treated in alcoholic solution with a strong base, such as potassium hydroxide or potassium ethoxide, in presence of chloroform, iodoform, or formaldehyde, to give by condensation on itself or with added quinoline or phenanthroline alkyl halide a carbocyanine dyestuff which is of value as an infra-red sensitiser for photographic emulsions. For example, β-naphthaquinaldine ethiodide is treated with alcoholic potassium hydroxide containing iodoform, when the corresponding carbocyanine dyestuff separates. It sensitises emulsions down to 740$\mu\mu$. T. S. WHEELER.

Manufacturing a perylene vat dye. A. ZINKE and H. SHOEPFER, Assrs. to F. BENSA (U.S.P. 1,569,111, 12.1.26. Appl., 17.10.23).—1 pt. of perylenequinone is chlorinated in about 20 pts. of nitrobenzene and the chlorine additive product formed, with or without separation from the nitrobenzene, is boiled for 10 min. with 2—10 pts. of aniline or other base, when hydrogen chloride is split off and chloroperylenequinone is formed. It separates on cooling in orange-red needles and dyes cotton in the vat fast brilliant greenish-yellow shades. T. S. WHEELER.

Vat dyes of the perylene series. COMP. NAT. DE MATIÈRES COLORANTES ET MANUF. DE PROD. CHIM. DU NORD RÉUNIS (ETABL. KUHLMANN), and H. PEREIRA (F.P. 589,643, 25.11.24. Conv., 30.11.23).—Bromoperylenes are treated with aromatic acids or their halogenides, anhydrides, or salts in the presence of condensing agents such as aluminium chloride. For example, dibromoperylene, m.p. 276—278°, when heated with benzoic acid and aluminium chloride at 150—170° gives a reddish-violet vat dye for cotton; bromoperylene, m.p. 189°, and benzoyl chloride give a blue dye. Dibromoperylene with benzoyl chloride gives a blue, with salicylic acid a brownish-red, with phthalic

acid a brownish-violet, and with naphthoyl chloride a reddish-violet dye. Dinitroperylene with benzoic acid gives a brown dye, with salicylic acid, a greenish-brown. The dye from chloroperylene and benzoic acid is reddish-violet. A. COULTHARD.

Vat dyes. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (F.P. 593,117, 9.2.25. Conv., 16.2 and 27.10.24).—Dyes are formed by condensing *p*-hydroxyphenazines, aminophenazines, arylaminophenazines, alkylaminophenazines, or naphthasultam-3:4-phenazines with 1:4-quinones (containing no halogens) or with *o*-hydroxy-1:4-quinones in the presence of substances which combine with water. The dyes formed in this way, may be oxidised with sodium hypochlorite if required. The dye (brown needles) from chloronaphthasultam-3:4-phenazine and 1:4-naphthaquinone dyes cotton and wool in yellowish-brown shades from the vat; treatment with sodium hypochlorite gives a greenish-yellow vat dye. Naphthasultam-3:4-phenazine and benzoquinone give a dye, brownish-red prisms, which dyes cotton in khaki shades. Treatment with hypochlorite results in a dye suitable for the production of lakes. The dye from α-naphthol-3:4-phenazine and benzoquinone, green needles, dyes cotton and wool in fast green shades, that from α-naphthylamine-3:4-phenazine and 1:4-naphthaquinone dyes cotton yellow. The dye from α-naphthol-3:4-phenazine and β-naphthaquinone gives greenish-yellow shades on wool and cotton. Similar dyes are obtained with β-naphthaquinone and naphthasultam-3:4-phenazine, 2-chloro-α-naphthol-3:4-phenazine, or phenyl-α-naphthylamine-3:4-phenazine. A. COULTHARD.

Manufacture of vat [benzanthrone] colouring matters. J. Y. JOHNSON. From BADISCHE ANILIN U. SODA FABR. (E.P. 247,261, 6.11.24).—See F.P. 589,323 ; B., 1926, 148.

Green sulphurised dyestuff. E. REBER and J. FRÖLICH, Assrs. to SOC. OF CHEM. IND. IN BASLE (U.S.P. 1,568,622, 5.1.26. Appl., 9.7.23).—See E.P. 201,540 ; B., 1924, 976.

Compounds for dyeing (U.S.P. 1,567,731).—See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of alkali with special application to wool. H. R. HIRST and A. T. KING (J. Text. Inst., 1926, 17, T 94—100).—Since many faults in woollen goods are due to retention of alkali, the determination of the latter is important. It is shown that common analytical methods are inadequate, but that wool gives up the whole of its alkali, whether in the form of hydroxide, carbonate, or soap, to terephthalic acid in aqueous suspension. The terephthalic acid which goes into solution can be determined by precipitation and weighing, but this method, though suitable for special cases of dyed wools, gives somewhat high results. Titration with bromophenol-blue as indicator is more convenient, and the authors prefer to add an excess of acid to the sodium terephthalate filtrate, filter, and titrate with sodium hydroxide. Calcium soaps also react with terephthalic acid, so a separate determination of calcium is necessary when they are present. J. C. WITHERS.

Determination of sulphuric acid in wool. H. R. HIRST and A. T. KING (J. Text. Inst., 1926, 17, T 101—103).—The wool is wetted and added to a measured volume of standard sodium terephthalate solution. The unchanged terephthalate is titrated in the filtrate (cf. preceding abstract). A better method for dyed wool, which is easily stripped, is to leave the material in a suspension of magnesium carbonate, filter, and precipitate the sulphate formed as barium sulphate. J. C. WITHERS.

Tensile strength and elongation of artificial silk. P. KRAIS (Z. angew. Chem., 1926, 39, 196).—The tensile strength of a single fibre of artificial silk (viscose) is reduced by 25% in a damp atmosphere (humidity 86%) compared with a dry atmosphere (humidity 52%), while the elongation before breaking is increased by 30%. The figures are 40% and 20% respectively when the fibre is completely soaked in water. For a strand composed of 27 fibres, the loss in tensile strength of the wet silk is 57%, while no change occurs in the elongation. B. W. CLARKE.

Banana fibre. R. O. BISHOP (Malay. Agric. J., 1925, 8, 376—381).—Fibre from the stem of cultivated varieties of banana is superior to that from the native wild varieties, and although the yield is relatively small the quality resembles that of medium grades of "partially-cleaned" manila hemp. D. F. TWISS.

Chemical examination of fibre from the bark of Terap, Tutor, and Baru. R. O. BISHOP (Malay. Agric. J., 1925, 8, 382—386).—The fibres from Tutor (*Artocarpus Kunstleri*, King), Baru (*Hibiscus macrophyllus*, Roxb.), and Terap (*H. floccosus*, Mast.) barks have similar chemical characteristics; the last, however, has a longer ultimate fibre and after treatment could probably be used as a substitute for hemp; the first two are alike in microscopic features and appear suitable for making paper of good quality. D. F. TWISS.

Action of concentrated sulphuric acid on cellulose and extraction of cellulose from wood. K. ATSUKI and T. MINAKI (Cellulose Ind., Tokyo, 1926, 2, 3—10; cf. B., 1926, 8).—Sulphuric acid at concentrations between H_2SO_4,H_2O and $H_2SO_4,3H_2O$ dissolves cellulose at the ordinary temperature without charring. Electrical conductivities of such hydrated forms indicate that the water is not entirely in combination. Dissolution of the cellulose is started by the combination of its OH groups with the sulphuric acid, followed by swelling and peptisation of the fibre. The rotation–time curve of the solution shows two points of inflection, as in the case of hydrochloric acid (*loc. cit.*), and the viscosity decreases rapidly until the first

point of inflection and then remains nearly constant as the cellulose changes from colloid to crystalloid products. The cellulose recovered by dilution is in a highly hydrated condition, drying to a horny product, and the amount obtained after 30 min. contact is over 100%. The quantity recovered by dilution decreases and its copper value increases at quicker rates the higher the concentration and temperature of the acid. α-Cellulose may be extracted from wood by treatment with 64—66% acid at 10—15° or with 66—70% acid at 0—10°, with but little loss. The wood, however, must be very finely powdered and passed through sieves of 64—120 meshes per sq. cm., and the precipitation of the cellulose by dilution must take place as soon as possible after solution. Japanese spruce yielded 45·2% of α-cellulose with copper value 1·30, free from furfuroids but sometimes slightly contaminated with lignin. J. F. BRIGGS.

Producing cellulose from bamboo. G. FESTER and G. MAIDANA (Z. angew. Chem., 1926, 39, 75—76).—Cellulose may be obtained by boiling bamboo from the Argentine Republic under pressure with 2·5% sodium hydroxide or a dilute solution of causticised wood ashes. The yield is about 45%, and it bleaches to a good white and is free from lignin. A. GEAKE.

Alcoholysis of wood. B. HOLMBERG and S. RUNIUS (Svensk Kem. Tidskr., 1925, 37, 189—197; Chem. Zentr., 1926, I., 136—137).—If the cellulose and lignin in wood are combined to form an ester, with lignin as the acid, treatment with an ethoxide or with alcohol and an acid will produce ethoxylignin, which will be saponifiable with alkali. If the lignin-cellulose compound is an acetal, it will not be decomposed by ethoxides. Treatment with alcohol and acid will produce lignin, if this is the alcoholic component; or an unsaponifiable ethoxylignin, if the lignin is the carbonyl component of the acetal. Experiments with a coniferous wood favoured the last theory. A. GEAKE.

Bleaching wood celluloses. H. WENZL (Papier-Fabr., 1926, 24, 81—88).—Hypochlorous acid oxidises more rapidly than does hypochlorite ion, and strongly hydrolysed hypochlorites, such as those of zinc and aluminium, are therefore more active than calcium hypochlorite. When cellulose is bleached in a hollander at 35° the product obtained with sodium hypochlorite has a lower ash content and copper number and a higher proportion of α-cellulose than that obtained with magnesium and, especially, calcium hypochlorite. The hydrate copper number is higher, which may be due to a greater swelling power of the sodium salt. It is possible that the impurities dissolve more readily as sodium than as calcium salts. The pentosan content is nearly the same in each case. Neutral salts may be expected to have an adverse effect on bleaching by diminishing the solubility of impurities. Cellulose bleached with a bleaching powder containing 34·6% of active chlorine and 2·4% of chloride chlorine is whiter than when bleached with a powder containing 25·4% of active chlorine and 9·2% of chloride chlorine. The white produced by sodium hypochlorite is adversely affected by additions of sodium chloride, sodium hydrogen carbonate, aluminium sulphate, or magnesium chloride. The addition of calcium carbonate to the bleaching powder used results in a higher copper number and pentosan content. When cellulose is bleached with sodium hypochlorite the active chlorine diminishes less rapidly than when the calcium salt is used. The addition of sodium carbonate to sodium hypochlorite greatly decreases the rate at which it is reduced, whereas sodium hydrogen carbonate and sodium hydroxide have only a small effect. The rate is also diminished by sodium and aluminium sulphates and potassium chlorate and nitrate, and is increased by calcium chloride, magnesium sulphate, and sodium acetate; sodium and magnesium chlorides have little effect. The addition of sodium acetate, and probably salts of other lower fatty acids, renders the bleaching inefficient. A. GEAKE.

Oxycellulose. H. KAUFFMANN (Textilber., 1925, 6, 591—592; Chem. Zentr., 1926, I., 536—537).—The depth of dyeing of material with Methylene Blue does not depend on its oxycellulose content; goods containing oxycellulose dye equally deeply after this has been completely removed by boiling. Oxycellulose is determined by measuring the "boiling-off number," and the "oxygen number." The former is the number of c.c. of 0·1*N*-potassium permanganate reduced by the organic substances extracted from 1 g. of the material by boiling with aqueous sodium hydroxide. The latter is the number of c.c. required to oxidise 1 g. of the material to carbon dioxide and water. It is concluded that oxycellulose has the formula $C_{12}H_{20}O_{15}$. In the hypochlorite bleach the most active constituent is free hypochlorous acid. A. GEAKE.

Polysaccharides, XXXIV. Hydro- and oxycelluloses. P. KARRER and T. LIESER (Cellulosechem., 1926, 1, 1—6).—Cotton wool is completely dissolved when it is steeped in an 84% solution of phosphoric acid for several hours at the ordinary temperature and then heated to 35° for ½ hr. By the addition of water a hydrocellulose is precipitated which is completely soluble in 10% sodium hydroxide, and which has a copper number (Schwalbe) of only 3. This hydrocellulose is readily methylated in cold sodium hydroxide solution by methyl sulphate. After four methylations a maximum methoxyl content of 42·5% is attained. As in the cases of normal cellulose (Irvine and Hirst, B., 1923, 710 A) and lichenin (Karrer and Nishida, B., 1924, 373) this value cannot be exceeded, although the theoretical value is 45·5%. This methylated hydrocellulose behaves like methylated lichenin in that it dissolves in cold water and is reprecipitated on boiling, and in being difficult to acetylate. The alkali-solubility of hydrocellulose is not due to carboxyl groups, and no alkali is used up; it is probably a case of peptisation or of the formation of additive compounds with alkali hydroxides. The electrical conductivity of the hydroxide is somewhat reduced. Oxycelluloses may be distinguished from hydrocelluloses by their acidity, but there is no

relation between the acidities of oxycelluloses and their copper numbers. The phosphoric acid hydrocellulose does not yield *iso*saccharic acid when boiled with lime. If oxycellulose is separated by dilute sodium hydroxide into soluble and insoluble portions, it is only the former that yields *iso*saccharic acid. Cellulose xanthate solutions may be purified by dialysis and the solution thus obtained contains 1 atom of sulphur and 1 of sodium for each 6 carbon atoms. The pure solution is not stable and when kept the cellulose is almost completely precipitated. The colours of the precipitates obtained with heavy metal salts are somewhat different from those given by impure solutions, and are as follows: copper, brown, turning yellow; bismuth, lemon-yellow; lead, white, turning yellow to brown; nickel, yellow, becoming greenish; iron, yellow. A. Geake.

Determination of lignin. S. Venkateswaran (J. Indian Chem. Soc., 1925, **2**, 253—260).—Before determining the lignin in straw it is necessary to extract the finely powdered substance for 3—4 hrs. with ether in order to remove all fats and resins. The absorption of phloroglucinol by the straw is about $2\frac{1}{2}$ times as great as that of the lignin isolated from it. Experiments show that this difference is due to the presence of furfuraldehyde-yielding constituents in the straw and not to loss of lignin by decomposition during extraction. For the isolation and determination of lignin the following method is recommended: 1 g. of ether-extracted straw is hydrolysed for 20 hrs. at room temperature with 15 c.c. of 72% sulphuric acid. The acid is diluted to 3% concentration and the solution boiled under reflux for 1 hr. The lignin is filtered off, washed, dried at 105°, weighed, and ignited. The hydrochloric acid method of hydrolysis, though satisfactory, is not so convenient as the sulphuric acid method, and Wenzl's method (B., 1924, 464) is unsatisfactory as the reagent attacks the lignin molecule. R. W. West.

Determination of the acidity of paper. S. Kohler and G. Hall (Paper Ind., 1925, **7**, 1059—1063; Chem. Abstr., 1926, **20**, 286).—The undesirable "internal" acidity (due to aluminium sulphate employed as rosin precipitant) and the harmless "external" acidity (due to aluminium sulphate added to preserve the glue) are determined by shaking 5 g. of the ground material with 250 c.c. of boiling water in small portions, heating for 1 hr. on the steam-bath under an air condenser, and repeating the extraction twice after filtration at the pump. After cooling, the extracts are separately titrated, using phenolphthalein, with 0·01*N*-sodium hydroxide. The "external" acidity alone is determined by shaking 10 g. in small cut pieces for 2 min. with 100 c.c. of cold water, filtering at the pump without washing, and titrating as before. A. A. Eldridge.

Removal of printers' ink from old paper. E. Berl and W. Pfannmüller (Z. angew. Chem., 1925, **38**, 887—889).—Experiments were made on the removal of printers' ink from old paper by shaking the shredded paper with a mixture of water and an organic liquid. Since cellulose is hydrophile and lampblack is hydrophobe a separation by flotation was to be expected. Trials were made with benzene, machine oil, carbon tetrachloride, and tetralin, the last-named proving the most satisfactory. Better results were obtained by combining a physical and chemical method of separation. The paper was first boiled with an excess of dilute sodium hydroxide solution and then treated with the water and tetralin. T. S. Wheeler.

Effect of dyes used in paper-making on animal life in water. Haempel.—See IV.

Patents.

Producing plastic masses from cellulose derivatives. P. Balke and G. Leysieffer (E.P. 229,674, 18.2.25. Conv., 18.2.24).—Plastic masses are produced by the process described in E.P. 154,157 (B., 1922, 383 a) but replacing cellulose derivatives by waste materials containing cellulose derivatives, which may be combined with gelatinising agents, *e.g.*, celluloid or cellulose acetate waste. A. Geake.

Manufacture of viscose solution. Maschinenbau-Anstalt Humboldt (G.P. 418,056, 3.12.21).—Cellulosic material is dissolved in the usual solvents (*e.g.*, zinc chloride, a mixture of strong acids, concentrated hydrochloric acid and a bisulphate, or zinc chloride and a bisulphate), separated from insoluble matter, precipitated, and washed. The purified cellulose is dissolved in the calculated amount of dilute caustic alkali and converted into viscose. Concentrated solutions may thus be obtained, suitable for the manufacture of plastic masses, artificial filaments, films, etc. A. Geake.

Manufacture of artificial threads, films, and the like from viscose. N. V. Nederlandsche Kunstzijdefabr. (G.P. 421,506, 18.1.22).—Carbon disulphide is added to ripened or unripened viscose solution and the mixture spun at once or after further ripening. A. Geake.

Manufacture of threads, films, and the like from viscose. H. Voss (G.P. 421,800, 7.11.22).—The precipitating bath consists of neutralised sulphite-cellulose waste liquor and ammonium salts. The latter may be added as such, or may be produced in the waste liquor or the precipitation bath. A. Geake.

System for boiling cellulose. Zellstoff-fabr. Waldhof, A. Schneider, and C. Hangleiter (E.P. 239,191, 15.8.25. Conv., 26.8.24).—In regenerating waste heat and gases (sulphur dioxide) from sulphite-cellulose boilers, by blowing the gases into fresh sulphite liquor (cf. G.P. 350,471; B., 1922, 855 a), undesirable back-pressure effects are avoided by a system of injectors, pumps, and valves. The gases are drawn from the boilers by pumping sulphite liquor through injectors, the vacuum chambers of which are connected to the tops of the boilers. The liquor then passes through a

further pump and check valve into a storage vessel. The liquor may be withdrawn from the bottom of the vessel for further circulation, or fresh liquor may be used continuously. A. Geake.

Plant where heat-consuming apparatus operates with a number of feed conduits. [Utilising heat accumulators in connexion with cellulose digesters.] J. Ruths, K. Ingerö, and H. Schrenk, Assrs. to Aktiebolaget Vaporackumulator (U.S.P. 1,568,420, 5.1.26. Appl., 7.12.22).—A cellulose digester is connected with the low-pressure steam supply (heat accumulator), but as soon as the pressure in the digester equals that in the low-pressure main a valve opens automatically and admits steam from the high-pressure main while communication with the low-pressure main is cut off. In this way the best use is made of the low-pressure steam, although the pressure at which it is supplied may vary, and little supervision is required even when operating a large number of digesters. T. S. Wheeler.

Treating seed hulls [to produce paper pulp]. R. Morgenier (U.S.P. 1,570,389, 19.1.26. Appl., 7.12.20).—Rice hulls are boiled for 2 hrs. with sodium hydroxide of 10° B. (*d* 1·07), the liquor is removed, and the residue beaten to a pulp. A. Geake.

Manufacture [drying] of paper. F. Grewin (E.P. 223,927, 24.10.24).—The drying is assisted by blowing air from the outside into spaces enclosed between the frame of the machine, the web of paper or cardboard and the dryer felt. The air is introduced through nozzles, which may be alternately on either side of the machine, and passes at a high velocity through the defined spaces in such a manner and direction that it does not impinge on the paper web but acts rather as an ejector for the accumulated water vapour. J. F. Briggs.

Paper making process. E. P. R. Marks. From Drying Systems, Inc. (E.P. 246,048, 10.8.25).—The moist sheet or web is partly dried by blowing steam into or through it while supported by wire gauze or other suitable porous material. Water is mechanically expelled and the web is heated ready for further drying operations. The steam may be supplied from a box contained in part of the suction roller drawing the web from the pulp vat. A. Geake.

Washing artificial silk. A. Pinel (U.S.P. 1,573,859, 23.2.26. Appl., 26.4.23).—See E.P. 199,355; B., 1924, 330.

Drying of textile and similar materials. H. L. Julien (E.P. 247,783, 14.4.25).

Manufacture of paper. [Pulping waste paper.] S. Leicester and L. B. Holman (E.P. 247,638, 13.11.24).

Tanning preparations from sulphite-cellulose waste lye (U.S.P. 1,571,873).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Use of reduction methods of analysis, particularly determination of the copper number, in determining bleaching damage. H. Wenzl (Woch. Papierfabr., 1925, 56, 994—997, 1024—1027; Chem. Zentr., 1926, I., 272).—The copper number of bleached cotton material is not an accurate criterion of the deterioration produced by bleaching unless considered together with tests of tensile strength. The solubility of oxycellulose increases with the alkalinity of the bleaching liquor. The most rapid and trustworthy method for determining copper number is that of Schwalbe and Hägglund (Fehling's solution is used), and those modifications involving the use of ferric sulphate or simplified apparatus (cf. Knecht and Thompson; B., 1920, 718 A) are less satisfactory. The changes which occur when Fehling's solution is heated may be very much reduced by the use of pure reagents and apparatus by which the overheating of the solution is avoided. A. J. Hall.

Comparison of the effects of oxidation before and after the mercerisation of the cotton fibre. E. Knecht and E. F. Muller (J. Soc. Dyers and Col., 1926, 42, 46—53).—The investigations were carried out on bleached American 2/50's cotton yarn, oxidation being effected by means of potassium permanganate (cf. Knecht and Thompson; B., 1920, 718 A) and bleaching powder, and mercerisation by means of caustic soda of 50° Tw. (*d* 1·25) and nitric acid of 83° Tw. (*d* 1·415) without tension. Cotton yarn mercerised with caustic soda shrinks less, becomes weaker, and has a smaller copper number and less affinity for dyestuffs than yarn mercerised by means of nitric acid; mercerisation increases the copper number of cotton. Mercerised cotton is more susceptible to oxidation and deterioration by exposure to heat and light than ordinary cotton, the yarn mercerised with caustic soda being more susceptible. Dyed mercerised yarns fade more rapidly than similarly dyed ordinary cotton, and this property is shown to a greater extent with cotton mercerised with caustic soda. Cotton yarns mercerised by caustic soda and nitric acid after either a moderate or high degree of oxidation suffer a decrease and a slight increase in textile strength respectively, but whereas nitric acid increases the copper number of the oxidised yarn slightly, caustic soda, owing to its solvent action, decreases the copper number considerably. Yarns weakened by oxidation suffer less than their usual shrinkage when mercerised with nitric acid. The loss of tensile strength produced by mercerisation of oxidised cotton with caustic soda decreases as the degree of oxidation is decreased and *vice versa,* and when the degree of oxidation is less than 0·0064 atom of oxygen per $C_6H_{10}O_5$ unit, mercerisation results in an increase of tensile strength, thus confirming the conclusion of Micheli (B., 1903, 1290). The ultimate effect of mercerisation with caustic soda is the resultant of weakening and strengthening actions due to solvent action and mercerisation respectively.

Although mercerised cotton yarns after oxidation with potassium permanganate have considerably higher copper numbers than similarly oxidised ordinary yarn, these differences do not occur when oxidation is carried out *in vacuo.* Under similar conditions, mercerised cotton is oxidised more quickly than ordinary cotton. Successive determinations of the copper number of the same sample of oxidised yarn yielded a a series of diminishing values whose aggregate totals in the case of mercerised (with caustic soda or nitric acid) and ordinary cotton subsequently oxidised to the same degree of oxidation, become nearly equal; the copper numbers were determined by the method of Knecht and Thompson (*supra*). Cotton yarns mercerised by means of various strengths of caustic soda and subsequently oxidised, have copper numbers which decrease as the strength of the mercerising liquor decreases. Approximately equal rises of temperature occur during the mercerisation of cotton with caustic soda and nitric acid under similar conditions. The affinity of mercerised cotton for oxygen and for moisture varies with the degree of mercerisation. The drying of mercerised yarn decreases its susceptibility to oxidation, but mercerised yarn maintained moist by physical methods shows no increased affinity for oxygen. The amount of moisture retained by completely mercerised yarn corresponds to the formula $2C_6H_{10}O_5,H_2O$. A. J. HALL.

Application of nitrobenzyl cellulose ethers to the dyeing of cotton. D. H. PEACOCK (J. Soc. Dyers and Col., 1926, 42, 53).—Cotton is dyed in shades fast to washing by esterification with *m*- and *p*-nitrobenzylphenyldimethylammonium chloride (Leucotrope is phenylbenzyldimethylammonium chloride), subsequent reduction of the nitro-group and then diazotisation and coupling with suitable components. For example, 1 g. of cotton boiled for 10 min. with 100 c.c. of a 1% solution of the nitro-derivative of Leucotrope containing a slight excess of sodium carbonate, is washed and reduced by treatment with an aqueous solution of sodium hyposulphite at 60—70°, is then diazotised at 5—7° in the usual manner and developed by immersion in aqueous solutions containing β-naphthol (for a rose shade), *R*-salt (rose), Schaffer's acid (red), *H*-acid (violet), chromotropic acid (red-violet), *m*-phenylenediamine (orange-brown), *J*-acid (red), phenyl-*J*-acid (red), carbonyl-*J*-acid (orange-red), γ-acid (acid coupling, red; alkaline coupling, bluish-red), or phenyl-γ-acid (violet-brown). A. J. HALL.

Comparison of the affinity of cotton, wool, and particularly cellulose acetate silk for azo-compounds [dyes] containing sulphonic, carboxyl, arsinic, and stibinic acid groups. A. J. HALL and M. I. AISCHE (J. Text. Inst., 1926, 17, T 104—110).—In the hope of discovering dyes soluble in water, but applicable to cellulose acetate without the agency of soaps or other solubilising substances, some azo-compounds containing arsinic or stibinic acid groups have been prepared. The dyes, the properties of which are summarised in a table, were prepared by diazotising and coupling from sulphanilic, *p*-aminobenzoic, *p*-arsanilic, and *p*-stibanilic acids. None of the compounds was suitable as a dye for cellulose acetate silk, but the results indicate that it should be possible to prepare azo-dyes containing arsinic and stibinic acid groups capable of satisfactory application to cellulose acetate. The stibinic acids are not fast to light, but the other dyes are about equally fast. The affinity for cellulose acetate increases in the order; sulphonic, arsinic, stibinic, carboxylic; and β-naphthol derivatives are more effective than α-naphthol compounds. J. C. WITHERS.

Bleaching wood cellulose. WENZL.—Sec V.

PATENTS.

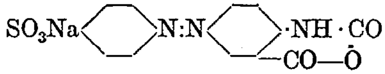
Bleaching hair, bristles, and the like. B. VENEZIANI (Austr. P. 100,709, 31.3.24).—Bristles are treated with hydrogen peroxide solution and then made alkaline with gaseous ammonia. The treatment may be repeated after drying, and the material may be subjected to a vacuum before the first treatment to remove air bubbles. A. GEAKE.

Rendering mercerised cotton, ammonium cuproxide [cuprammonium] silk, viscose silk and like products refractory against the further absorption of direct dyes. CHEM. FABR. VORM. SANDOZ (E.P. 241,854, 9.1.25. Conv., 24.10.24).—The processes previously described (cf. G.P. 346,883 and E.P. 233,704; B., 1922, 214 A; 1925, 956) for preparing "immunised" cotton (cf. E.P. 246,609; following abstract) are similarly used for immunising mercerised cotton and cellulose artificial silks, suitable esterifying agents being the chlorides and anhydrides of aliphatic and aromatic carboxylic acids, and chlorides of aromatic sulphonic acids. For example, immunised viscose silk is obtained by immersing 1 kg. of viscose silk for 5 min. at 20° in a 10% alcoholic solution of caustic soda, centrifuging, then immersing the silk for 5 min. at 65° in 10 kg. of a 20% solution of acetyl chloride in toluene, afterwards centrifuging and soaping, washing, and drying at 50°. Similarly to immunise or mercerise cotton, 1 kg. of the cotton is immersed for 30 min. at 20—25° in 10 kg. of a 14% alcoholic solution of caustic soda, then centrifuged, immersed for 2 hrs. at 40—45° in 10 kg. of a 20% solution of *p*-toluenesulphochloride in toluene, again centrifuged, then soaped, washed, and dried at 50°. A. J. HALL.

Dyeing of [" immunised "] cotton materials. SANDOZ CHEMICAL CO., LTD., and A. E. WOODHEAD (E.P. 246,609, 17.11.24).—"Immunised" cotton (*i.e.*, cotton resistant to direct dyestuffs) prepared by treatment of ordinary cotton with alcoholic caustic soda and *p*-toluenesulphochloride by the method described in E.P. 195,619 (cf. E.P. 233,704; B., 1925, 956) is dyed by means of insoluble or difficultly soluble dyestuffs which have been solubilised by pre-treatment with suitable dispersing agents; *e.g.*, higher fatty acids or their sulpho-derivatives, their alkali-metal or ammonium salts. The methods of dyeing are similar to those generally used for

cellulose acetate except that the temperature of dyeing is preferably 75—95°. Claim is made for use of the following dyestuffs: azo dyestuffs prepared from nitroanilines, dinitroanilines, nitrotoluidines, nitroxylidines, anisidines, chloroanisidines, nitroanisidines, phenetidines, chlorophenetidines, nitrophenetidines, or chloronitroanilines diazotised and coupled with aniline, methylaniline, dimethylaniline, ethyl- or di-ethylaniline, methylethylaniline, diphenylamine, methyl- or ethyl-diphenylamine, *m*-toluidine, or *p*-xylidine; aniline, chloroanilines, dinitroanilines, toluidines, xylidines, nitrotoluidines, nitroxyhdines, anisidines, chloro- or nitro-phenetidines diazotised and coupled with α- or β-naphthylamine or their methyl, dimethyl, ethyl, diethyl, methylphenyl, or ethylphenyl derivatives; α- or β-naphthylamine or their chloro- or nitro-derivatives diazotised and coupled with aniline or the alkyl- or aryl-aniline or naphthylamine derivatives mentioned above, or α- or β-naphthylamine; naphthols, resorcinol, β-hydroxynaphthoic acid and its anilide, toluidide, and similar derivatives coupled with diazotised amino-bases such as *m*-nitroaniline and α-naphthylamine. The method of dyeing allows the production of two-colour effects in union fabric containing ordinary cotton and other fibres, together with immunised cotton. A. J. HALL.

Compound for dyeing. BADISCHE ANILIN- U. SODA-FABR., Assees. of F. GÜNTHER (U.S.P. 1,567,731, 29.12.25. Appl., 8.6.25).—An aromatic *o*-aminocarboxylic acid other than an uncoloured anthranilic acid derivative is treated with carbonyl chloride or ethyl chloroformate to give a derivative of isatoic anhydride which combines with cellulose or other carbohydrates forming a kind of ester of an aminoarylcarboxylic acid so that the dyed substance is a part of the dye molecule. If suitable groups are present the carbohydrate compounds can be coupled with diazo-compounds or diazotised and coupled with azo-dye components. For example diazotised sulphanilic acid is coupled with anthranilic acid and the product is treated with carbonyl chloride in feebly alkaline solution to give a substance with the formula



which dyes cellulose in an alkaline bath yellow shades. If the coloured fibre is diazotised and coupled with β-naphthol a bright red shade results. 2:3-Naphthylaminecarboxylic acid and carbonyl chloride yield a compound which when combined with cellulose gives no colour but the fibre can be diazotised and coupled with azo-dye components. If ethyl chloroformate is used it is boiled with the substance under treatment. T. S. WHEELER.

Apparatus for the liquid treatment of textile materials upon rollers. J. BRANDWOOD, A. STOCKER, and TWYVER WORKS, LTD. (E.P. 247,661, 21.11.24).

Carrying out and controlling reactions. (G.P. 422,159).—See I.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Gypsum–sulphuric acid process (of the Farbenfabr. vorm. F. Bayer & Co.). W. J. MÜLLER (Z. angew. Chem., 1926, 39, 169—174).—Calcium sulphate in the form of gypsum or anhydrite is reduced by carbon in the form of coke, at 800° according to the equation $CaSO_4+C=CaO+SO_2+CO$. The lime must be recovered in a marketable form to make the process economically possible, and clay is therefore added to the raw materials in the requisite proportions to obtain Portland cement. The operation is carried out in a rotary kiln fired with pulverised fuel. The materials must be dried before entering the kiln in order to obtain a sufficiently high concentration of sulphur dioxide in the issuing gases. An oxidising atmosphere must be maintained in the kiln to prevent the formation of carbon oxysulphide in the gases and calcium sulphide in the cement clinker. In practice, about half the theoretical quantity of coke is used, to prevent too rapid reduction and the formation of calcium sulphide in the clinker. The Portland cement produced is of good quality and is ground with blast-furnace slag and sold as blast-furnace cement. The gases from the kiln contain 6—7% SO_2; after electrical separation of dust and washing, they are used to make sulphuric acid, by the contact or chamber process. B. W. CLARKE.

Determination of hydrocyanic acid in gaseous mixtures. G. E. SEIL (Ind. Eng. Chem., 1926, 18, 142—143).—The gas containing hydrogen cyanide is caused to react with 0·1*N*-iodine solution in the presence of starch solution saturated with sodium hydrogen carbonate in a special 500-c.c. Tutwiler burette. The hydriodic acid is thus neutralised as formed. The iodine is added until the starch solution just turns blue, and the liquid is is then withdrawn from the burette, which is washed down with distilled water or sodium hydrogen carbonate solution. Standard sodium thiosulphate solution (1 c.c.=0·2288 mg. per litre of HCN gas on a 500-c.c. sample) is used for exact titration of the excess of iodine and enough hydrochloric acid is then added to give an acid reaction (amount previously ascertained), whereby cyanogen iodide and hydriodic acid interact to yield hydrocyanic acid with liberation of iodine. If a blue colour appears after keeping for 0·5—1 minute hydrocyanic acid is present, and the solution is titrated to the point of disappearance of colour. The difference between the two burette readings ×10 gives the grains of hydrocyanic acid per 100 cub. ft. from 500 c.c. of sample. D. G. HEWER.

Determination of strength of liquid hydrocyanic acid by specific gravity. M. WALKER and C. J. MARVIN (Ind. Eng. Chem., 1926, 18, 139—142).—The strength of hydrocyanic acid solutions may be rapidly determined to an accuracy within 0·2% by taking the specific gravity by means of hydrometers with a scale from 0·650 to 0·750,

with 50 divisions on each, extending over a stem length of 106·4 cm., so graduated that readings may be taken to the third decimal place. The liquid is drawn up by suction into a glass cylinder containing the hydrometer and the thermometer passes through the cylinder stopper. The table of strengths of acid corresponding to specific gravities given was prepared by determinations on liquids of different strengths analysed by the gravimetric method of absorption in caustic soda and titration with silver nitrate. D. G. HEWER.

[Manufacture of solid] calcium cyanide. F. J. METZGER (Ind. Eng. Chem., 1926, **18**, 161—163).—Calcium cyanide, previously known only in dilute solution, is now being manufactured in the solid state by the action on calcium carbide of liquid hydrogen cyanide containing 2% of water calculated on the weight of carbide. $CaC_2+4HCN=Ca(CN)_2,2HCN+C_2H_2$. The reaction is carried out in a jacketed vacuum filter to which the carbide is gradually added from a gas-tight hopper with cooling. The acetylene evolved contains a small proportion of hydrogen cyanide, which is removed by washing with alkali. On completion of the reaction the sludge is warmed, the hydrogen cyanide distilling off and the calcium cyanide being obtained as a light tan-coloured powder which is immediately filled into airtight containers. This product, which contains about 55% of cyanogen, is very rapidly decomposed by atmospheric moisture, with liberation of hydrogen cyanide, and it has been termed "powdered hydrocyanic acid." It offers many advantages over liquid hydrocyanic acid in the fumigation of citrus trees against scale and in other similar applications. C. IRWIN.

Nitrogen fixation by means of barium carbonate and coal in an electric oven. A. SCHWEITZER (Z. Elektrochem., 1926, **32**, 98—105).—A prepared mixture of barium carbonate and coal was coked and then treated with nitrogen in a graphite crucible heated to 1300°. An absorption of 6·6% of nitrogen was obtained. The product, consisting of 3 pts. of barium cyanide and 1 pt. of barium cyanamide, was treated with water in an autoclave at 7—8 atm. pressure for ¼ hr. The gases, consisting of ammonia and hydrogen, were led through sulphuric acid, and the ammonia was determined. In the technical process, the finely powdered coal is mixed with barium carbonate, a little coal tar and water, and formed into cylindrical pieces in a hydraulic press. The coking takes place in a retort oven at 800° for 1½ hr. For the nitrogen absorption an electric furnace of special construction is used, having graphite electrodes, single-phase alternating current being employed at 45—65 volts. Great economy of power may be effected at this stage, which can be further improved by additional protection against loss of heat. The heated mass is treated first with producer gas, and later with 99% pure nitrogen. The reaction product is treated with steam at 7 atm. pressure in an autoclave twice, the gases being collected in sulphuric acid. The nitrogen absorption is about 4%. The explanation of the reactions adopted is that the barium carbonate becomes reduced first to oxide and then to carbide, which reacts with nitrogen giving barium cyanide or barium cyanamide and carbon. The stoichiometric relations require 1 mol. $BaCO_3$:4C, but it is better in practice to use carbon greatly in excess, in the approximate proportion 1 mol. $BaCO_3$: 18C. The action of water on barium cyanide and barium cyanamide yields ammonia in each case, together with barium formate and barium carbonate respectively. The thermal decomposition of barium formate yields hydrogen and barium oxalate; the latter on further heating yields barium carbonate, which is thus recovered for subsequent operations. C. H. D. CLARK.

Technology of calcium cyanamide. P. DOLCH (Z. Elektrochem., 1926, **32**, 68—70).—The method of determining carbide in a finely-powdered sample by dropping saturated salt solution upon it mixed with salt and examining the evolved gas is replaced by that of adding the carbide in small quantities at a time to water. The error in the older method lies in the retention of acetylene in the lime sludge, in a polymerised form. This was proved by evaporating the ether extract, when a tarry residue remained. No decomposition of carbide occurs during grinding. The influence of foreign gases in lessening the nitrogen absorption by finely-milled carbide in a heated oven is very considerable. The efficiency of the process is greatly increased by sweeping out foreign gases periodically by means of a current of nitrogen. If in a series of ovens the outgoing gas from one oven passes into the next, the proportion of foreign gases leaving each oven becomes progressively greater, with corresponding decrease of nitrogen absorption. Hydrogen is formed during the heating by decomposition of acetylene. C. H. D. CLARK.

Hydration of lime. W. G. WHITMAN and G. H. B. DAVIS (Ind. Eng. Chem., 1926, **18**, 118—120).—The effects of the hydration of lime at various temperatures with water vapour and with water in amounts up to 20 times the theoretical quantity upon the quality of the hydrated product have been studied by means of the rate of reaction of the product with acid, its rate of settling in water, and its appearance under the microscope. In general, the quality as judged by any one of these methods agrees closely with the ratings obtained by the other two. Hydration with the theoretical amount of water or with water vapour tends to produce excessively high temperatures locally in the lime, thus delaying the completion of the reaction and allowing time for the agglomeration of the particles with the result that a low-grade product containing coarse grains is formed. The three most important factors are the ratio of water to lime, the temperature of hydration, and the degree of agitation, the best product being obtained by hydrating with a large excess (20 times the theoretical amount) of water in boiling solution. A. W. HOTHERSALL.

Fusion of alumina with soda [sodium carbonate]. G. FELD (Z. angew. Chem., 1926, 39, 174—175).—Bauxite or aluminium hydroxide precipitated from aluminium chloride by ammonia, when fused with sodium carbonate at 900—1200°, gives the sodium aluminate Na_2O,Al_2O_3. Aluminium hydroxide precipitated by carbon dioxide from a solution of sodium aluminate, at temperatures below 950° forms the aluminate $2Na_2O,Al_2O_3$, which at higher temperatures is converted into the aluminate Na_2O,Al_2O_3. The different behaviour of these various forms of alumina is assumed to be due to a difference in structure of the hydroxides. B. W. CLARKE.

Quick method for the determination of ozone. H. B. McDONNELL (Ind. Eng. Chem., 1926, 18, 135).—Low concentrations of ozone may be measured by allowing the ozonised air to flow from a nozzle with an outlet of definite size (*e.g.*, 1·5 mm.) through a glass tube into a testing solution consisting of potassium iodide and starch, and an amount of sodium thiosulphate solution equivalent to 5 c.c. of 0·01% iodine solution. The gas is passed until the blue colour appears and the time noted with a stop-watch. The method is also applicable to dilute mixtures of chlorine and air. D. G. HEWER.

See also A., Mar., 261, **Determination of sulphites and of ferrous iron** (BONNER and YOST); **Volumetric determination of sulphates** (ATKINSON). 263, **Use of uranous sulphate in volumetric analysis** (VORTMANN and BINDER).

Determination of perchlorate in Chile saltpetre. HOFMANN, HARTMANN, and HOFMANN.—See XVI.

PATENTS.

Production of phosphoric acid. CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 241,903, 20.10.25. Conv., 23.10.24).—Phosphorus pentoxide fume, which is very difficult to condense, is easily dissolved by water or phosphoric acid solution near its boiling point. To produce phosphoric acid, phosphorus or a mixture containing it may be burnt in a tower down which water trickles, the heat of combustion of the phosphorus heating the water to the required temperature to dissolve the fume. E. S. KREIS.

Manufacture of sulphuric acid. W. CARPMAEL. From F. BAYER U. Co. (E.P. 245,903, 5.12.24).—In the manufacture of sulphuric acid by the combined contact process and chamber process, the amount of contact catalyst is reduced to $\frac{1}{6}$—$\frac{1}{10}$ of that ordinarily used. The sulphur dioxide is transformed into sulphur trioxide by the usual contact process and the remaining sulphur dioxide is converted into sulphuric acid by the chamber process, *e.g.*, as described in E.P. 149,648, 187,016, and 184,966 (cf. B., 1921, 693 A; 1922, 982 A, 858 A). E. S. KREIS.

Production of hydrocyanic acid. E. EICHWALD (F.P. 595,399, 18.6.24).—Catalysts for use in the production of hydrocyanic acid from gas mixtures containing nitrogen and carbon compounds, *e.g.*, from ammonia and carbon monoxide, consist of metal compounds containing oxygen, which, under the conditions of the reaction, are not, or are only partially reducible, *e.g.*, they may contain ferric oxide and alkali or alkaline-earth oxides. The catalysts are preferably deposited upon material such as pumice stone, asbestos, or wood charcoal. L. A. COLES.

Mechanical salt-cake furnaces. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 225,548, 24.11.24. Conv., 29.11.23).—A mechanical salt-cake furnace, in which the process is completed in one stage, is constructed of acid-resisting brick or stone, instead of cast iron. It is preferably fired with producer gas with recovery of the waste heat. The advantages are greater durability and the possibility of the use of higher temperatures. C. IRWIN.

Separating a mixture of hafnium and zirconium compounds. N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 226,180, 20.11.24. Conv., 12.12.23).—A mixture of the phosphates, phosphites, arsenates, arsenites, antimonates, or antimonites of hafnium and zirconium is introduced into a medium containing hydrofluoric acid or an alkali bifluoride. The complex acids or their salts produced are then fractionally crystallised. The ammonium salts are preferred, but examples are also given of the use of lithium and of barium salts. C. IRWIN.

Making chlorides [aluminium chloride]. G. L. PRITCHARD and H. HENDERSON, Assrs. to GULF REFINING Co. (U.S.P. 1,568,181, 5.1.26. Appl., 17.11.21).—A mixture of bauxite or other aluminous material and coke is introduced into the top of a heat-insulated tower divided into two sections. In the upper section it is preheated to 560° by the direct or indirect action of furnace gases and in the lower it is subjected to the action of chlorine or hydrochloric acid. Aluminium chloride is formed and volatilises, passing to a condenser by an exit pipe at the top of the lower section. Means are provided for passing the material from the upper to the lower section while preventing gas from passing in the opposite direction, and also for impeding the fall of the material down the tower. To provide the heat necessary to complete the formation of aluminium chloride, a small quantity of aluminium dross may be added to the material to be treated. T. S. WHEELER.

Recovering borax from saline liquors. A. W. GAUGER and H. H. STORCH, Assrs. to BURNHAM CHEMICAL Co. (U.S.P. 1,571,002, 26.1.26. Appl., 21.2.25).—The liquor is evaporated until other salts crystallise, leaving a concentrate of metaborate and tetraborate. This is cooled and agitated in contact with a finely divided material, when crystals of borax are rapidly formed. C. IRWIN.

Production of alkali cyanides. STOCKHOLMS SUPERFOSFAT FABR. AKTIEBOLAGET, Assees. of H. G. A. RAMSAY (Swed. P. 58,030, 23.2.24).—Mixtures of calcium cyanamide with alkali chlorides or alkali carbonates, or with both, are fused and

treated with moderately concentrated alkali hydroxide solution, preferably hot, in quantity at least sufficient to convert the whole of the calcium into calcium hydroxide. After filtration, the solution is evaporated to recover alkali cyanide. L. A. COLES.

Manufacture of oxygen. SOC. L'OXYLITHE (E.P. 240,412, 3.7.25. Conv., 25.9.24).—The coil for liquefying the oxygen in the vapours emanating from the liquid air surface is placed outside the rectifying column instead of in the lower part of the column, thus simplifying construction and allowing free trickle of liquid in the column. E. S. KREIS.

Detecting oxygen. A. H. MAUDE, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. CO. (U.S.P. 1,567,534, 29.12.25. Appl., 2.1.24).—A glass cylinder contains a known volume of a standard solution of indigo-carmine reduced with a standard solution of alkaline stannite, the space above the reagent being evacuated. The cylinder is sealed below by a closed capillary tube. The space under test, for example, the gas space of a transformer, is connected to the capillary tube and the latter is broken, whereupon gas from the transformer bubbles up through the liquid in the container into the evacuated space. The time required for the liquid to turn from orange to green gives a measure of the oxygen present. The solution indicated is suitable for gases containing 1—21% of oxygen. For smaller percentages ammoniacal cuprous chloride is used. Other gases can be similarly determined, using suitable reagents.

T. S. WHEELER.

Purification of radium emanation. V. F. HESS and E. D. LEMAN, Assrs. to UNITED STATES RADIUM CORP. (U.S.P. 1,570,834, 26.1.26. Appl., 14.5.23).—The emanation, after a preliminary partial purification, is passed into a tube containing an absorbent capable of removing the remaining impurities. The part of the tube containing the impurities is then sealed off from the remainder which contains purified emanation. An apparatus for carrying out the process comprises an explosion chamber, an expansion chamber, an absorption bulb, an auxiliary trap chamber, a capillary tube, and a vacuum pump with the necessary connexions.

C. IRWIN.

Treatment of earthy minerals. W. FELDENHEIMER (U.S.P. 1,573,385, 16.2.26. Appl., 26.5.25). —See E.P. 242,358; B., 1926, 55.

Recovery of caustic hydroxides from waste solutions. F. H. GRIFFIN, Assr. to VISCOSE CO. (U.S.P. 1,573,703, 16.2.26. Appl., 27.6.23).—See E.P. 237,468; B., 1925, 757.

Carrying out and controlling reactions (G.P. 422,159).—See I.

Drying and calcining gypsum (U.S.P. 1,571,189). —See IX.

Portland cement and sulphuric acid (F.P. 596,423).—See IX.

Electrolysis of alkali chlorides (F.P. 596,918). —See XI.

VIII.—GLASS; CERAMICS.

Action of molten glass on refractory materials. O. BARTSCH (Glastech. Ber., 1925, 3, 249—265; Chem. Zentr., 1926, I, 1469).—Pots are actively attacked, but for a short time only, by the batch materials, and the attack of the glass itself is milder, though of longer duration, being a process of solution. For this reason glazing of the walls and rapid melting are necessary. The vitreous ground mass of the clay passes into the glass, and the mullite structure of the wall grows. The quaternary eutectic of composition SiO_2 65·27%, Al_2O_3 16·98%, CaO 5·08%, K_2O 12%, melts at 1180° and may pass into the glass. Still greater amounts of alumina can be dissolved, but this is opposed by the slow rate of solution. In the bounding layer between pot and glass a highly aluminous, viscous mass is formed containing mullite and sometimes corundum, especially in the case of highly aluminous clays, and this layer protects the walls. If the density of the bounding layer deviates from that of the melt, the layer tends to move either to the top or bottom, in which case further pot surface is exposed, whilst the upward movement tends to produce hard cords. For this reason clays low in alumina are specially suitable for pots. Bond clay and grog should have the same composition. Cavities in the pot body increase the possibility of attack. Homogeneous pots are most readily prepared by casting. A. COUSEN.

Silica bricks. Thermal analysis. TRAVERS and DE GOLOUBINOFF (Rev. Mét., 1926, 23, 27—47).— The thermal expansion of the various forms of silica was investigated using the dilatometer designed by Chevenard. The test-pieces were prepared by moulding cylinders of the finely-powdered material mixed with 3—4% of bonding material (potassium silicate or lime) and slowly heating to 800°. The results obtained with quartz confirm those of Le Chatelier except that the reversible expansion at 575° is found to be 0·27, Le Chatelier's figure being 0·42. A new point in the tridymite curve at 440—450°, is evidence of an undescribed δ-tridymite. Above 440°, the expansion of tridymite is very small but is always positive. Very pure cristobalite gave a greater thermal expansion than that used by Le Chatelier, the total expansion between 20 and 210° being 1·55, and the expansion at 210° being 1·05, Le Chatelier's figures being 1·2 and 1·00 respectively. The transformation $\alpha\rightarrow\beta$-cristobalite is shown to be rapid and reversible.

P. B. ROBINSON.

PATENTS.

Glasses. JENAER GLASWERK SCHOTT UND GEN. (E.P. 219,972, 25.7.24. Conv., 3.8.23).—A series of glasses are claimed which all contain as ingredients SiO_2, Al_2O_3 up to 10% (if present), B_2O_3 from 5 to 18%, K_2O and Na_2O together from 3 to 15%, the Na_2O not exceeding 1% and being at most one-fifth of the percentage of the K_2O. They possess the advantages that they can be worked for optical purposes

without devitrifying or becoming turbid, whilst they have a high thermal endurance. Such glasses have a small dispersion and a comparatively high refraction. Other ingredients of the glasses may be (1) PbO, Sb_2O_3 forming together with the K_2O and Na_2O from 3 to 35% of the glass; (2) BaO, CaO, MgO, ZnO, which together with the K_2O and Na_2O form at most 35%; (3) PbO, Sb_2O_3, BaO, CaO, MgO, ZnO, which together with the K_2O and Na_2O form at most 35% of the glass. A. Cousen.

Cord-free glass. Comp. des Lampes (F.P. 594,354, 23.1.25. Conv., 25.1.24).—Cord-free glass for optical purposes is prepared within a vertically-arranged cylindrical container, which either consists entirely of a platinum–iridium alloy or of tungsten with an inner lining of platinum–iridium alloy. The upper portion of the cylinder is widened to a funnel shape, the lower part narrowed. The truly cylindrical portion is provided with two superimposed heating-wire coils, the narrow portion with a third heating coil. In the upper portion of the cylindrical tube is a stirring device, movable in a vertical direction. Beneath the exit opening is a dish capable of rising and falling in order to receive molten glass. The apparatus is filled to the upper rim with the batch, which is melted in the cylindrical part, with stirring. The lower portion of the cylinder is more strongly heated, and then the exit freed by the melting of the batch. When sufficient glass has flowed out the opening is again closed by discontinuing the heating. A. Cousen.

Tunnel ovens for ceramic and chemical purposes. H. T. Padelt (E.P. 245,005, 16.6.25).—A tunnel oven has mixing chambers for gas and air disposed in rows one above the other and side by side in the oven wall, and air and gas flues having separate control devices open into the mixing chambers. The mixing chambers and burners are arranged in groups having separate gas and air flues. The gas supply may be increased or decreased at will in accordance with the temperature and output required, and the regulation can also be so performed as to obtain a higher or lower temperature at the higher or lower part of the tunnel as required. P. B. Robinson.

Melting magnesite-sand. C. Fiedler (G.P. 421,428, 24.7.24. Conv., 26.7.23).—The sand is bonded with a magnesium chloride cement and the pieces, together with natural lump magnesite, are fired in suitable furnaces. A. Cousen.

Stone- or porcelain-like mass. H. Morin-Krop (Swiss P. 103,400, 13.11.22; G.P. 421,422, 13.11.23).—The material consists of 84—78·5% SiO_2, 1·54—1·47% Al_2O_3, 5·85—5·5% CaO, 1·13—1·02% K_2O, and 7·26—13·5% Na_2O. It is obtained by melting a mixture of sand, a natural fluoride (as fluorspar), a double silicate of aluminium (felspar), and soda ash. It is highly acid-resisting and a good electrical insulator. A. Cousen.

Red glazes and enamels. Patent-Treuhand Ges. für Elektrische Glühlampen, Assees. of H. Nachod (G.P. 421,424, 27.5.24).—Red glazes and enamels are prepared by adding chromates, particularly dichromates, and one or more opacifying agents to a basic glass. A layer of the glaze 0·05 mm. thick is coloured, by this means, red throughout. A. Cousen.

Fuel feeding arrangement for tunnel furnaces. J. B. Monnier (E.P. 232,929, 10.3.25. Conv., 25.4.24).

IX.—BUILDING MATERIALS.

Aluminous cements. G. Agde and R. Klemm (Z. angew. Chem., 1926, **39**, 175—186).—Bauxite from the Vogel mountains can be used as a raw material for aluminous cement, the chemical composition of the product falling well within the range of these cements on the Gibbs triangular system. Microscopical examination shows the cements to consist of an amorphous ground-mass together with crystals of melilith; on setting, similar effects are observed to those obtained with Portland cement, the amorphous material being attacked first by the water and converted into a mass of interlocking crystals which probably account for the quick-setting properties of the cement. Subsequently, lime and alumina go into solution, and then colloidal alumina and silica are deposited in a reactive "gel" form, which is converted into calcium silicates, aluminates or aluminosilicates, thus causing the hardening of the cement. A range of products can be obtained in which a gradual transition from aluminous to Portland cement occurs. Materials with a very high lime content appear to set very rapidly, but develop no subsequent strength. Replacement of a large proportion of the alumina in aluminous cements by ferric oxide reduces the strength, since calcium ferrite has no binding powers. Aluminous cements are very resistant to the attack of acids (10% potassium bisulphate solution), unless the iron content is high, and to magnesium sulphate solutions. B. W. Clarke.

Design of concrete mixtures. D. A. Abrams (Bull. I., Struct. Materials Res. Lab., Lewis Inst., Chicago, 1918. Revised 1925. 20 pp.).—The strength of concrete depends on the ratio of water to cement used, an increased water-ratio giving decreased strength. To produce concrete of a given strength with the minimum of cement, the aggregate must be selected and graded to produce a workable concrete with the minimum quantity of water. The "fineness modulus," *i.e.*, the sum of the percentages by weight of the material retained by successive sieves in the Tyler standard series, forms a rational basis for proportioning the aggregate. Tables are given for determining the relative proportions of fine and coarse aggregate required to produce an aggregate of the desired "fineness modulus," which will determine the consistency of the concrete used. Concrete of a given strength, *i.e.*, water-ratio, may therefore be produced by an infinite variety of mixtures of fine and coarse aggregate and cement, and the selection of the mixture to be used will depend on the degree

of plasticity or workability required, cost and availability of various types of aggregate, and other properties desired. Thus maximum density of concrete (*i.e.*, lowest percentage of voids) is not always produced by the grading of aggregate giving maximum strength, this discrepancy being greater for richer mixes. Tables and charts are given whereby the best concrete mixture can be designed for any given conditions. B. W. CLARKE.

Blast-furnace slag as a building material. R. GRÜN (Deuts. Bergwerks.-Ztg., Festnummer Bauwesen u. Baustoffind. Reprint. 4 pp.).—Brief descriptions are given of the preparation and use of crushed slag, slag sand (granulated slag), slag stone, slag cement (from granulated slag and lime), and iron Portland cement and blast-furnace cement (from slag and Portland cement clinker). B. W. CLARKE.

Mercuric chloride [corrosive sublimate] for timber impregnation. R. FALCK and S. MICHAEL (Z. angew. Chem., 1926, 39, 186—193).—Mercuric chloride, although apparently taken up in large amounts by timber, is in reality only adsorbed by the wood substance, and does not form any chemical compound with the wood. The mercuric chloride can be recovered almost quantitatively as such by extracting the wood with water, and the use of mercuric chloride for timber preservation is therefore useless in exposed conditions from the point of view of durability and also dangerous. Further, owing to the adsorption of the sublimate by the fibres, the penetration into the wood itself is very small, the mercuric chloride remaining concentrated on the surface. A mixture of sodium fluoride and mercuric chloride does not give improved results, since no chemical compound is formed analogous to that obtained with sodium chloride and mercuric chloride, and the constituents therefore react independently. Further, the mercuric chloride is liable to be reduced in contact with metal fittings etc. to mercury and mercurous chloride, which has no poisonous or disinfectant properties.
B. W. CLARKE.

Gypsum-sulphuric acid process. MÜLLER.—See VII.

PATENTS.

Bituminous mixtures for road construction etc. UNIVERSAL RUBBER PAVIORS (MANCHESTER, 1923), LTD., and A. E. BROWN (E.P. 246,186, 4.4.25).—Clay, ground so that 95—98% passes a 200-mesh sieve, and dried at 300—400° to remove hygroscopic water, is agitated relatively slowly with bitumen at 80—100°, forming a colloidal dispersion of the clay, which is not liable to settle out on storing and is suitable as a grouting or covering material for road work and the like. B. W. CLARKE.

Bituminous mixtures for road construction. UNIVERSAL RUBBER PAVIORS (MANCHESTER, 1923), LTD., and A. E. BROWN (E.P. 246,065, 4.4.25. Addn. to 246,186; preceding).—Residual products of gutta-percha, balata, or low-grade rubber-like materials, *e.g.*, gutta-percha pitch, are added to bituminous mixtures prepared as described in the original patent, forming a composition which adheres well to concrete and road surfaces, and is stable under all conditions of climate and traffic.
B. W. CLARKE.

Bituminous compositions. W. J. MELLERSH-JACKSON. From G. C. WARREN (E.P. 246,548, 22.10.24).—Rubberised fabric is mixed with mineral aggregate (gravel, crushed stone, etc.) at a temperature (120—200°) sufficient to soften the rubber, and after agitating violently a molten bituminous cementing material is added (generally at 120—180°) and thoroughly amalgamated. The rubber gives resiliency and the fibres of the fabric give strength and durability to the composition, which can be used for road surfacing, flooring, roofing, and the like.
B. W. CLARKE.

Making [non-adhesive] bituminous emulsions. L. KIRSCHBRAUN (U.S.P. 1,567,061, 29.12.25. Appl., 17.1.21. Renewed 31.3.24).—In the preparation of emulsions of asphalt dispersed in water containing colloidal clay (cf. U.S.P. 1,302,810; B., 1919, 494 A) the tendency of the clay to surround the asphalt particles and render the whole non-adhesive is increased by addition of an acid flocculating agent such as aluminium sulphate or sodium hydrogen phosphate. T. S. WHEELER.

Drying and calcining gypsum. C. DRESSLER, Assr. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,571,189, 2.2.26. Appl., 25.2.20).—The material is transported through a long horizontal heating chamber, terminating at one end in a depending portion, by a number of conveyor belts arranged longitudinally one above the other, so that at the discharge end the upper belt projects beyond the belts beneath it and at the opposite end the lower belt projects beyond the belts above it.
B. W. CLARKE.

Manufacture of Portland cement and sulphuric acid. F. G. GOETZMANN (F.P. 596,423, 3.4.25).—Gypsum or anhydrous calcium sulphate is mixed with clay, sand, and a small quantity of coal, and the mixture so finely ground that, at the most, 8% of residue is left on a 4900-mesh per sq. cm. sieve. The mixture is slightly moistened and then heated at 1400—1500° in a rotary kiln. The sulphur dioxide evolved is converted into sulphuric acid.
A. COUSEN.

Cement from blast-furnace slag. R. GRÜN (G.P. 421,776, 11.3.23).—A cement resistant to salt water is made by grinding blast-furnace slag with highly aluminous clinker or with fused aluminous cement in the same proportions as are usual with blast-furnace and iron–Portland cements. The products are sufficiently resistant for all building purposes and they are more resistant to salt water, and cheaper than aluminous cement. A. COUSEN.

Magnesia cement. FARBENFABR. VORM. F. BAYER UND CO., Assees. of H. VON KÈLER and J. DRUCKER (G.P. 421,987, 21.2.24).—An aqueous

suspension of crystalline magnesium chloride or an aqueous solution of the chloride is mixed with magnesia in desired proportions and the resulting powder is heated to about 250°. The magnesium chloride solution and magnesia may be mixed by simultaneous spraying and the mixture dried by hot gas. Bonded magnesia cement or materials prepared from magnesia cement may be regenerated by heating to somewhat above 250°. A. Cousen.

Refractory cement and concrete. P. J. F. Kestner (U.S.P. 1,573,072, 16.2.26. Appl., 12.3.25). —See E.P. 231,141; B., 1925, 719.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Production of iron from silicates and pyrites. G. Tammann and G. Bätz (Z. anorg. Chem., 1926, **151**, 129—139).—When hornblende or basalt is mixed with wood charcoal and a small proportion of lime or sodium carbonate and heated first at 600° and then at 1400°, the iron is reduced almost quantitatively. In the absence of alkali, or when heated directly to 1400°, the reduction is less complete, and it is further diminished by the addition of quartz. When heated with precipitated and ignited silica, ferric oxide, magnetite, and ferrous oxide each begins to react at 800°, but reaction with quartz does not begin below 950°. More heat is evolved with the silica than with quartz. Solid ferric oxide and magnetite do not react directly with silica, but only after loss of oxygen and formation of ferrous oxide; this begins at 800°. When iron silicates, prepared by heating ferric oxide and silica, are reduced with hydrogen for one hour at 850°, the proportion of the iron reduced falls from 24·4% when the molecular fraction of silica is 0·375 to 0·3% when it is 0·600. When the silica content is further increased the proportion of the iron reduced rapidly increases, and is 38·4% when the molecular fraction of silica is 0·920. When iron pyrites is heated a halt in the heating curve occurs at 684°, due to loss of sulphur. In the presence of lime no halt is observed; the liberated sulphur combines with the lime to form calcium sulphate and sulphide. A mixture of pyrites and lime is only slightly reduced when heated with wood charcoal. By the interaction of lime and pyrites a difficultly reducible calcium iron oxysulphide is formed, and only a small quantity of ferrous oxide. A. Geake.

Influence of alternating currents on the electrolytic corrosion of iron. A. J. Allmand and R. H. D. Barklie (Faraday Soc., Feb., 1926. Advance proof).—Experiments on the corrosion of iron in alkaline solutions by direct current confirmed those of other workers, the presence of chlorine ions activating the metal. When alternating current was used, change in the alkali concentration of the electrolyte had no particular influence, but lowering the current frequency produced an increased corrosive effect, and there seems little doubt that the discharge of hydrogen ions during the cathodic impulse activates the iron by decreasing the oxygen content in the surface layer of metal. The effect of alternating current superposed on direct current was a marked increase in corrosion, and where an anode remained passive either at a *D.C.* intensity of 0·125 amp./cm.^{2} or at an *A.C* intensity of 0·375 amp./cm.^{2} and 400 frequency, the anode became definitely yellow in two minutes under the combined effect. A high *AC:DC* ratio (more than 2) was favourable to corrosion, as was also an increase in current density and low frequency. Typical subsoil drainage liquid saturated with carbon dioxide yielded similar results. C. A. King.

Widmannstätten structure in iron–carbon and iron–nickel alloys and in meteorites. T. Kasé (Sci. Rep. Tôhoku, 1925, **14**, 537—558).—Hypoeutectoid steel gives the Widmannstätten structure on annealing above 1000° or on heating it to partial melting and cooling in air from a temperature above the Ar3 point. Hypereutectoid steel must be cooled from above the Acm point. Quenching is not an effective method and cooling slowly through the A1 transformation point destroys the characteristic structure. The Widmannstätten structure is obtained in iron–nickel alloys by a long annealing above the Ac3 point and then quenching in water, and the formation of this structure in meteorites may be due to a long exposure to a very high temperature and subsequent rapid cooling. C. A. King.

Self-magnetisation of steels under torsion. R. Cazaud (Compt. rend., 1926, **182**, 467—468).—A study of the self-magnetisation of steels under torsion suggests that such measurements may be used to obtain rapid indications of composition and of thermal treatment. Test-pieces of steels containing from 0·3 to 1·0% of carbon were placed, under torsion, in the magnetic field of a doubly-wound bobbin. With a constant current flowing through the primary winding of the bobbin, any deformation of the test-piece through torsion causes a variation of the magnetic flux, and sets up induced currents, recorded photographically, in the secondary coil. Torsion produces a negative variation of the magnetic flux in mild steels containing less than 0·5% of carbon, and a positive variation with steels in which the carbon content is more than 0·5%. With extra hard steels, cast steels, and certain special steels, of martensitic structure, the variation of the flux with torsion is continuous. Repeated torsions cause the steels to approach a state of magnetic saturation. Cold working tends to increase the variations produced in the magnetic flux by torsion. F. G. Tryhorn.

Specific volume determinations of carbon and chromium steels. J. H. Andrew, M. S. Fisher, and J. M. Robertson (J. Roy. Tech. Coll. Glasgow, 1925, [2], 70—78).—The specific volume of steels containing up to 1·2% C increases as the temperature of quenching is raised to an extent which is greater the higher the carbon content. This is evidently due to expansion of the martensite. With more than 1·2% C austenite is produced in amounts which increase with rise of quenching temperature so

that the specific volume of the steel begins to decrease again. This decrease is most marked after quenching from 1100°. If, however, the same steels are heated to 1100° for a short time, allowed to cool to 1000—800°, and then quenched, the specific volumes are extraordinarily high, possibly owing to graphitisation having taken place. The increase in specific volume on quenching indicates that martensite is a solid solution of cementite in ferrite in which the iron lattice has been expanded by carbon and that the amount of this expansion produced by a definite quantity of carbon in solution exceeds the volume of the corresponding quantity of cementite. The specific volume curves for chromium steels are similar to those for plain carbon steels. The effect of tempering chromium steels with more than 1% C is first to reduce slightly the specific volume then between 200° and 300° to cause it to increase rapidly corresponding with the tempering of the austenite ; above 300° simultaneous tempering of austenite and martensite results in a decrease in the specific volume. With a low-carbon chromium steel a steady fall in the specific volume takes place with rise in temperature of tempering. Austenitic carbon steels with or without chromium increase in specific volume after immersion in liquid air although the electrical properties remain unchanged. A. R. POWELL.

Crystalline structure of metals. J. H. ANDREW (J. Roy. Tech. Coll., Glasgow, [2], 63—69).—A speculative discussion in which the author attempts to correlate the atomic structure of metallic crystals with the physical properties of the metal, with especial reference to iron and steel. The part played by surface tension at the grain boundaries of a multicrystalline substance in determining the hardness is briefly discussed with reference to the volume changes on quenching and tempering martensitic steels (cf. preceding abstract). A. R. POWELL.

Magnetic properties of permalloy. D. BINNIE (J. Roy. Tech. Coll., Glasgow, 1925, [2], 5—7).—The initial permeability of annealed permalloy (78·5% Ni, 21·5% Fe) is 30 times that of the best soft iron and a field as low as that of the earth will saturate the alloy to a magnetic intensity comparable with that of soft iron. The magnetic properties are, however, very sensitive to strain, which causes a marked diminution of the susceptibility. Thus, a thin strip of permalloy after coiling and uncoiling exhibited magnetic properties similar to those of steel. A. R. POWELL.

Cementation of ferrous alloys by tungsten. J. LAISSUS (Compt. rend., 1926, 182, 465—467 ; cf. B., 1925, 635).—The action of finely powdered ferrotungsten as a cementing agent for electrolytic iron and for steels containing from 0·15% to 0·8% of carbon, has been studied. The temperature of cementation varied from 800° to 1100°, and its duration from 2½ to 10 hrs. Photomicrographs of sections showed the existence of two zones : an inner one of solid solution, well developed during the longer heats, and a bright outer layer, consisting probably of tungsten carbide. The thickness of the latter increased with the time and with rise of temperature The cementation zone decreased in thickness with increase in carbon content of the steel. Steels cemented with tungsten are more resistant to an oxidising atmosphere at 900° than uncemented steels, up to a period of 5 hrs. Beyond that time, the cemented layer rapidly disintegrates by scaling. The cemented steels have considerable resistance to corrosion in water and sulphuric and hydrochloric acids, but are very rapidly attacked by nitric acid. F. G. TRYHORN.

Determination of vanadium in ferrovanadium. K. SOMEYA (Sci. Rep. Tohôku, 1925, 14, 577—591 ; cf. B., 1925, 29).—The sample (0·5 g.) is dissolved in nitric acid, the excess expelled completely by evaporation with sulphuric acid, the insoluble material filtered off after addition of 50 c.c. of water, and the solution oxidised with permanganate, nearly neutralised with ammonia, cooled, and diluted to 120 c.c. A few drops of diphenylamine solution and an excess of ferrous sulphate are added, followed by potassium dichromate solution in excess of that required to oxidise the ferrous sulphate. After addition of 10 c.c. of concentrated hydrochloric acid and 15 c.c. of phosphoric acid mixture the excess of dichromate is titrated with ferrous sulphate. A. R. POWELL.

Wet method for the determination of silver in lead. B. S. EVANS (Analyst, 1926, 51, 79—81).—When cupellation is impracticable silver may be determined in lead by the following procedure : 50 g. of metal are dissolved in nitric acid, ammonia is added until the solution is alkaline, followed by acetic acid until just acid, and the liquid is boiled for 2 hrs. with a coil of copper foil. The precipitated silver and excess of copper are filtered off, washed with hot water, and redissolved in nitric acid. The solution is made just alkaline with sodium hydroxide and again acid with hydrochloric acid, adding a slight excess of the latter to precipitate all the silver. After boiling for 15 min., the solution is kept overnight and filtered. The precipitate is dissolved in 10—15 c.c. of 1:1 ammonia, the solution diluted to 200 c.c., an excess of standard potassium cyanide solution added, followed by a few crystals of potassium iodide, and the excess cyanide titrated with a standard silver solution as in the case of nickel. A. R. POWELL.

Constitution of the alloys of silver and tin. A. J. MURPHY (Inst. Metals, Mar., 1926. Advance copy, 18 pp.).—A re-examination of the equilibria in the system silver–tin by microscopical, thermal, and electrical resistance methods has confirmed the shape of the liquidus and the composition of the eutectic (96·5% Sn, m.p. 221°) as found by Petrenko (B., 1907, 615) but the constitution of the solid alloys differs widely from that found by earlier investigators. The solid solution of tin in silver contains a maximum of 13·3% Sn at 724° and this slowly falls to just below 11% at 100°. This α solution reacts with liquid at 724° to form a second solid solution, β, which is the sole constituent of alloys containing 12·5—19% Sn at 20° and of alloys containing 13—24%

Sn at 480° ; at 724° it contains 14·5% Sn. The $\alpha+\beta$ field is a narrow belt extending over the range 13·3—14·5% Sn at 724° and 11—13% Sn at 20°. The γ constituent is the compound Ag_3Sn, which exists in a narrow range of less than 1% about 26% Sn, the $\beta+\gamma$ field occupying the space between the β and γ ranges mentioned above. Alloys containing more than 27% Sn are all composed of γ and tin containing less than 0·1% Ag in solid solution. The α solid solution is characterised by prolific twinning accompanied by the development of finer marking following the course of cleavage planes in the crystals. The β solid solution consists entirely of polygonal crystals without any surface markings and the compound Ag_3Sn, after prolonged annealing, of polygonal crystals with fine cross-hatch markings. The inversion in this compound at 232° recorded by Petrenko has not been confirmed, but by electrical resistance measurements a transformation of unknown nature has been found to take place at 60°. The addition of even very small quantities of silver to tin suppresses both the allotropic transformations, 0·1% Ag preventing the tin from changing to the grey variety even after 3 weeks at –78°. A. R. POWELL.

Influence of gases on copper at high temperatures. I. A. G. LOBLEY and D. JEPSON (Inst. Metals, Mar., 1926. Advance copy, 13 pp.).—A special type of electric resistance furnace used in the experiments is described. The furnace can be evacuated or filled with any desired gas. The crucible containing the copper can be lowered from the heating zone into a water-cooled chamber, thus giving a rapid and reproducible rate of cooling. The behaviour of the copper while molten and during solidification was observed through observation windows in the top and side of the furnace. The charge consisting of 220 g. of pure copper was heated in nitrogen, hydrogen, and carbon monoxide at atmospheric pressure to various temperatures between the melting point (1083°) and the boiling point (2310°) for 30 min., after which the crucible was lowered into the cooling chamber and rapidly cooled. The resulting ingots were examined for blowhole volume (by apparent density), hardness, and macrostructure. The Brinell hardness numbers were in no case affected by the treatment. The period and intensity of ebullition on cooling were observed and the weight of copper thrown up as spray was determined, and from these data a confirmation was obtained of the relation between the amount of gas held in the cavities of the solidified ingot to the total amount driven out. Up to temperatures of 1900° and 1700° respectively, nitrogen and carbon monoxide are not absorbed by molten copper in excess of that soluble in the solid metal. Hydrogen is absorbed to a varying degree at all temperatures between the melting point and 2200°, and is ejected on solidification, forming blowholes. The blowhole volume–temperature curve indicates a fall from the melting point to a minimum at approximately 1350°, rising to a maximum at about 1750°, and then falling as the temperature rises to approximately 2200°, the last fall being probably connected with the increasing vapour pressure of the copper. A. W. HOTHERSALL.

Determination of zinc oxide in brass. B. S. EVANS and H. F. RICHARDS (Inst. Metals, Mar., 1926. Advance copy, 7 pp.).—Purified hydrogen is passed over a sample of the brass weighing about 80 g. at a temperature of 980° (at which the brass is molten but the volatilisation of zinc is not excessive), and the water formed is absorbed and weighed. The reverse reaction by which the water formed is reduced in the cooler parts of the furnace by the volatilised zinc is suppressed by the insertion on either side of the combustion boat of a tightly fitting copper gauze roll, 5 in. long, and a ball of copper wire rolled to fit the combustion tube. Both the rolls and balls are deoxidised prior to the determination by heating to 1000° for some hours in a current of hydrogen. The hydrogen gas is passed at a measured rate of about one litre per hour, both the blank and the actual test being continued until a constant increase in the weight of the absorption tube is obtained per litre of gas passed. The accuracy of the method was established by reduction of weighed amounts of zinc oxide, the preparation of a standard sample of brass containing a known amount of zinc oxide having been found impracticable. The amount of oxygen present in a series of commercial brass ingots was found to vary from 0·002% to 0·005%. A. W. HOTHERSALL.

Copper-rich aluminium–copper–tin alloys. D. STOCKDALE (Inst. Metals, Mar., 1926. Advance copy, 27 pp.).—The liquidus curve of aluminium–copper alloys falls to a minimum at 8·3% Al and then rises to a maximum at 12·5% Al (cf. B., 1922, 818 A); addition of tin reduces the melting points of these alloys in such a way that if the temperature is plotted against the atomic percentage of copper, the liquidus curve of any series of alloys containing a constant proportion of tin is nearly parallel to that of any similar series, *i.e.*, the minimum at 82 at. % Cu and the maximum at 75 at.% Cu are independent of the kind of foreign atom present. From these results it is shown by van't Hoff's law that the first solid to separate at the maximum contains no tin and that the latent heat of solidification of a solution of one atom of aluminium in three atoms of copper is the mean of its constituents, indicating that no combination to Cu_3Al takes place. Up to 9% Sn the eutectic line of the solidus becomes much depressed so that a valley runs through this part of the ternary model; on the left of this valley the α solid solution of aluminium and tin in copper separates out and on the right the β solid solution is formed from the liquid and α phases. The limit of the α solid solution at room temperature is very nearly a straight line on the ternary triangular diagram. The field of the β phase is rendered extremely complex by the addition of tin, decreasing progressively as the tin increases up to 9%. The eutectoid transformation temperature is raised considerably in the alloys richest in copper by adding tin, which also reduces the apparent velocity of the transformation. This change takes place at approximately constant

temperatures for alloys containing the same percentage of aluminium but varying amounts of tin, *e.g.*, the alloys containing 7% Al with 5, 7, or 9% Sn all undergo the transformation at 600—610°. Alloys comparatively rich in aluminium and tin and consisting of the β phase at high temperatures undergo complicated changes on cooling; at first the δ phase separates, but, on further cooling, this appears to redissolve in the remaining β or reacts with it to form a new phase which cannot be distinguished microscopically from β but which appears to be polymorphous with it. The phase changes on cooling an alloy with 9% Al and 9% Sn may be represented as follows:

$$\text{Liquid} \xrightleftharpoons{991^\circ} \text{liquid}+\beta \xrightleftharpoons{815^\circ} \beta \xrightleftharpoons{750^\circ} \beta+\delta \xrightleftharpoons{600^\circ} \beta' \xrightleftharpoons{555^\circ} \alpha+\beta'.$$

A. R. POWELL.

Mechanical properties at high temperatures of an alloy of nickel and copper with especial reference to "creep." H. J. TAPSELL and J. BRADLEY (Inst. Metals, Mar., 1926. Advance copy, 19 pp.).—Tensile, torsion, impact, fatigue, and hardness tests at various temperatures up to 800° are recorded for an alloy consisting of 68·74% Ni, 28·16% Cu, 2·35% Mn, 0·56% Fe, 0·10% C, 0·008% S. The ultimate stress falls slightly with increase of temperature from 37 tons per sq. in. at 15° to 33 tons per sq. in. at 400° and thereafter rapidly to 7·5 tons per sq. in. at 800°. The elongation is 46% at 15°, 38—35% at 100°—400°, 19% at 500°, 30% at 700°, and 20% at 800°; the reduction in area remains fairly constant at about 70% up to 300°, then falls rapidly to a minimum of 21% at 500°, increasing again to 30% at 700° and once more falling with further rise in temperatur, ewhereas the limit of proportionality falls steadily with increase of temperature. The limiting "creep" stress decreases at about the same rate as the ultimate tensile strength from 24 tons per sq. in. at 400° to less than 1 ton per sq. in. at 700°, while the modulus of rupture remains constant to 300°, then falls rapidly, and the resistance to impact of a notched bar falls slowly throughout the range. The impact hardness number remains nearly constant at about 234—224 up to 300°, then falls slowly to 185 at 400°, remaining constant at this figure up to 600° and falling to 151 at 700°. The limiting fatigue range is ±15 tons per sq. in. at 15° and ±11·7 tons per sq. in. at 500° and the mean coefficient of expansion between 0° and 800° is 0·000017. A. R. POWELL.

Sand-cast aluminium–manganese alloys. S. DANIELS (Ind. Eng. Chem., 1926, **18**, 125—130).—The properties of a number of sand-cast aluminium–manganese alloys containing up to 9·6% Mn have been investigated. The tensile strength was raised from 5·25 tons/in.^{2} for the alloy containing no manganese to 7·6 tons/in.^{2} with 1·02% Mn, but the elongation (on 2 in.) fell rapidly from 38·5% to 22·6% and then to 2·6% with no manganese, 1·02% and 2·88% Mn respectively. The Brinell hardness numbers showed a gradual increase from 20 to 48 with increasing manganese content. The presence of manganese increased the shrinkage and promoted porosity when present in appreciable quantity. The alloy containing 4·6% Mn machined like cast iron, probably owing to the presence of the hard and brittle compound $MnAl_3$. The alloys were readily attacked by distilled water (30 days, immersion), but showed only slight corrosion after 100 hrs. in salt spray. Heat treatment at 580° for 96 hrs. followed by quenching in water and ageing at 149° for 2 hrs. did not materially alter either the physical or mechanical properties or the corrodibility. The metallography is discussed and a number of photomicrographs are reproduced.

A. W. HOTHERSALL.

Physical properties of very light magnesium alloys. A. PORTEVIN and F. LE CHATELIER (Compt. rend., 1926, **182**, 382—384).—For magnesium alloys, the following formulæ express the specific volume (V) and electrical resistance (R) as functions of x (the percentage of added metal) $V=0{\cdot}575-K_Vx$ in cm.^{3}/g. and $R=4{\cdot}5+K_Rx$ in microhms/cm^3/cm. The values for K_V and K_R found for various added metals, the limiting values of which are given in brackets, are: Al (7%), 0·0022, 1·34; Cd (6%), 0·0047, 0·15; Cu (13%), 0·0052, 0·01; Mn (4%), 0·004, 0·13; Ni (15%), 0·00538, 0·06; Pb (5%), 0·0056, 0·58; Zn(5%) 0·0043, 0·28. For aluminium and zinc the values given in brackets represent the limiting solid solution and the limit of homogeneity respectively; in other cases they represent the limiting alloy examined. The coefficient of expansion is in no case greatly different from that of magnesium; zinc and lead cause an increase whilst copper, nickel, and especially silicon cause a decrease. Ternary alloys have also been investigated. Taking into account the mechanical properties it is concluded that the binary magnesium alloys with nickel and copper, and the ternary alloys with aluminium–nickel and aluminium–copper promise to be most useful. For certain applications such as motor pistons they give the most suitable values for density, elastic limit, hardness, heat conductivity, and expansion. R. A. MORTON.

Recent developments in metallurgical research. New facts developed by high-power metallography. F. F. LUCAS (J. Roy. Microscop. Soc., 1925, 393—404).—The examination at high magnifications of austenite containing a network of martensitic figures shows that the martensitic needle is always characterised by a central mid-rib or axis which etches darker with picric acid than the remainder of the needle. The directions of the axes of martensitic needles within any similarly oriented portion of an austenite grain indicate that martensite results from decomposition along the crystallographic planes of austenite. No case was found of a martensitic needle crossing a grain boundary or a twinning plane of austenite. Multiple twinning bands of the order of 0·00001 in. are often found in martensitic needles. Two varieties of troostite, nodular and flocculent, are recognised. The former may develop directly from austenite, or from an austenite–martensite matrix, in which case it has its origin in a martensitic needle either along the mid-rib or at the junction of two needles.

After the nodule has formed, the excess constituent is rejected to the grain boundaries and at the same time stratification sets in and the radial grain of the troostitic nodule is transformed into a grain of pearlite. Whereas nodular troostite responds to a test for crystallinity the flocculent variety does not, probably because the particles composing it are not within the range of resolution. Evidence has been obtained that the precipitation of ferrite marks the very early stages of the transformation of austenite and martensite into pearlite under definite conditions of heat treatment. The amount of flocculent troostite formed is a measure of the excess constituent present. A steel of eutectoid composition yields little or no troostite and the austenite and martensite form pearlite without passing through a troostitic stage. Photomicrographs at 3950 diameters magnification illustrate the different stages in the decomposition of austenite in a specimen of previously normalised 1% C steel which was heated to a high temperature at one end while the other end was maintained at room temperature, and the whole then quenched in iced brine. The effects of cold work and the mechanics of the process of regranulation by annealing were studied in a billet of permalloy (78·5% Ni, 21·5% Fe) cold-rolled from 1-in. to $\frac{3}{8}$-in. rod, one end of which was subjected to a long anneal and the other maintained cold. In addition to slip-bands, incipient fissures were observed. Twinned areas developed gradually by reorientation of distorted metal. Small areas within the same grain developed a more pronounced orientation towards the annealed end of the rod. At low magnification the large elongated grains produced by cold work persisted in phantom outline after the metal had regranulated on annealing.

M. Cook.

High-power metallography. F. F. Lucas (J. Franklin Inst., 1926, **201**, 169—216; cf. preceding abstract).—The application and limits of high magnification in metallurgical photomicrography are discussed. The process of the decomposition of austenite to pearlite in a 0·5% carbon steel is illustrated with photomicrographs at a magnification of 3230 diameters. The extreme and intermediate stages are obtained in one specimen by suitable heat treatment. The hardened end of the specimen consists of a network of martensitic needles in austenite. On approaching the abrupt transformation troostite appears along some of the martensitic needles and ferrite soon appears in the centre of the troostitic areas. As the troostite increases it forms a border enclosing areas of martensite and austenite. It continues to increase until it reaches a maximum and then decreases, leaving large areas of austenite and martensite. The central areas of ferrite increase with the troostite, and when no more of the latter is formed the ferrite increases at its expense until it disappears except for a small border surrounding large areas of ferrite. This residual troostite is of eutectoid proportions and stratifies, assuming the orientation of adjoining grains. At the same time small cubic pits develop in the austenite–martensite and increase in number along the cleavage planes of the austenite. These pits unite to form long bands of ferrite, showing that stratification takes place in austenite and martensite areas without these areas passing through troostite. The troostite which is formed is a rough measure of the excess ferrite present. Nodular troostite was obtained in 0·5% carbon steel quenched under uniform conditions. The phenomenon of regranulation on annealing after cold work is illustrated with photomicrographs at high magnifications.

M. Cook.

Liquation in molten alloys and its possible geological significance. S. W. Smith (Inst. Min. Met., Feb., 1926. Advance copy, 48 pp.).—The liquation phenomenon in molten alloys known as inverse segregation is discussed at length, and it is shown that this behaviour is determined by differences in temperature between different portions of a homogeneous molten mixture before solidification commences and that the constituent which liquates towards the cooling surface is that which, if added in further quantity to the mixture under consideration, would lower its freezing point. Reasons are also advanced for considering that the Ludwig–Soret effect in solutions is identical with liquation in molten alloys and that the liquation phenomena observed in alloys are applicable to molten matter in general. Preliminary experiments on copper matte containing 29—3% Cu have been carried out to test this theory and distinct evidence of the liquation of copper towards the chilled surface has been obtained. On the basis of these results the mode of primary copper enrichment in certain pyritic ore-bodies is discussed. A. R. Powell.

Volatilisation of lead during lead burning, using different flames. H. Engel and V. Froboese (Arch. Hyg., 1925, **96**, 69—101; Chem. Zentr., 1926, I., 1479).—Volatilisation of lead during such processes as lead burning and applying homogeneous lead coatings, takes place to the greatest extent with the oxy-hydrogen flame, using an excess of oxygen, but is reduced considerably by carburising the gas, or by using coal gas instead of hydrogen; it is a minimum when a pale, non-sooting acetylene flame is used. Decrease in the volatilisation when hydrocarbons are present in the gas appears to be due to the formation of a carbon monoxide–hydrogen zone in the flame. No lead hydride was detected in the combustion gases, the volatilised material appearing to consist principally of lead monoxide mist in a sufficiently fine state to remain in suspension in the air for a long time. L. A. Coles.

Composition of copper mattes. B. Bogitch (Compt. rend., 1926, **182**, 468—470; cf. B., 1926, 194).—Equilibrium conditions have been determined in the system Cu–S–Fe, in the liquid state, and at temperatures near the solidification point. Ferrous sulphide and iron are miscible in all proportions in the liquid state, but addition of copper, in amounts more than 3%, causes a separation into two layers, the upper high in sulphur content, the lower very low in sulphur content. As the amount

of copper is increased, most of the metal passes into the upper layer, until its copper content reaches about 50%. Copper added above this amount tends to collect in the lower layer, until this contains 94—95% Cu. Beyond this point, additional copper tends to collect in the upper layer. The limiting system Cu_2S–Cu, free from iron, consists, in the liquid state, of two conjugate solutions. The bearing of the phase diagram for the above system on the metallurgy of copper is discussed.

F. G. TRYHORN.

Successive potentiometric titration of copper and iron in metallurgical products. T. F. BUEHRER and O. E. SCHUPP, JUN. (Ind. Eng. Chem., 1926, 18, 121—124).—0·3—0·4 g. of the sample is dissolved in aqua regia and the solution is evaporated to dryness with excess of strong hydrochloric acid. The residue is digested with a minimum of water acidified with 1—2 c.c. of strong hydrochloric acid and the solution is filtered. The filtrate is acidified with 5—10 c.c. of strong hydrochloric acid and is covered with a layer about 1 in. thick of kerosene, previously purified by successive treatment with several charges of concentrated sulphuric acid. 10—15 c.c. of chromous chloride solution in excess of that required to turn the solution red with reduced copper are added, the reduction of both copper and iron being prompt and complete if the solution is kept agitated. The solution, the volume of which should be limited to 50—70 c.c., is titrated with potassium dichromate solution, galvanometer readings being taken at intervals of 0·1 or 0·2 c.c. around the inflection point, and the true end point obtained by interpolation. The chromous chloride solution, which is kept under a layer of kerosene, maintains a fairly constant normality whether in acid or nearly neutral solution or in contact with rubber. It is prepared by boiling potassium dichromate in hydrochloric acid solution with excess of zinc free from arsenic and iron until reduction to the light blue solution is complete. Quantitative and reproducible results were obtained for copper and iron simultaneously by this method and also by direct titration with chromous chloride solution, the time taken being less than half that ordinarily required. Reproducible results could not, however, be obtained either by direct titration with stannous chloride or with titanous chloride or by titration with potassium dichromate after reduction with stannous mercuric chloride (Penny's method for iron), stannous chloride alone, or with titanous chloride. Molybdenum, selenium, arsenic, and antimony might interfere with the method if present in considerable quantity. The method has not been applied to products very low in copper such as slags and tailings.

A. W. HOTHERSALL.

Critical studies on the fusion of rare metal ores. II. Separation of tantalum and columbium [niobium]. G. W. SEARS (J. Amer. Chem. Soc., 1926, 48, 343—348; cf. A., 1925, ii., 580).—A method is given for the separation of tantalum and niobium by the pyrosulphate fusion of tantalite and columbite. The temperature of fusion has a great influence on the subsequent solubility of the tantalum and niobium compounds, the best temperature for the separation by means of concentrated sulphuric acid (1 : 1 acid dissolves niobium completely, whilst the tantalum is left in the residue) being 835—875°. The proportion of pyrosulphate used in the fusion mixture and the time of heating have little or no effect on the solubility. Concentrated sulphuric acid is a more effective differentiating solvent than hydrofluoric acid. The compounds formed by the fusion are probably tantalates and niobates rather than sulphates or double sulphates.

W. THOMAS.

Corrosion of iron. W. G. WHITMAN (Chem. Reviews, 1926, 2, 419—435).

See also A., Mar., 229, **Electrical conductivity of silver–zinc alloys** (PETRENKO). 230, **Plasticity of amorphous and crystalline solids** (BECKER). 231, **Thermal expansion of tungsten** (HIDNERT and SWEENEY). 233, **Vapour tensions of mercury, cadmium, and zinc** (JENKINS). 248, **Cathodic pulverisation of alloys** (MAZUR). 254, **Solid metal–hydrogen compounds** (HÜTTIG). 261, **Electrometric determination of tellurium** (SCHRENK and BROWNING); **Determination of potassium as perchlorate** (YOE); **Determination of rubidium and cæsium** (STRECKER and DIAZ). 262, **Separation of lead and silver** (VORTMANN and HECHT); **Determination of thallium** (STRECKER and DE LA PENA); **Electrolytic separation of copper from cadmium** (JILEK and LUKAS); **Titration of mercury with potassium cyanide, lead with arsenate, and iron with thiosulphate** (JELLINEK and CZERWINSKI). 263, **Confirmatory test for aluminium** (ESTILL and NUGENT); **Separation of iron and aluminium from zirconium** (LESSING). 264, **Distillation of mercury containing gold** (RIESENFELD and HAASE).

Inductive heating. NORTHRUP.—See XI.

PATENTS.

Production of castings containing silicon. RHEINISCHE EISENGIESSEREI UND MASCHINENFABR. A.-G. (E.P. 237,554, 27.2.25. Conv., 26.7.24).—High-silicon iron castings are produced in preheated moulds as in the pearlite-casting process. The temperature of the moulds varies from 500° for a 10% silicon content to about 150° for a 20% silicon content, the temperature also being lower the greater the wall thickness.

T. H. BURNHAM.

Magnetic [iron–silicon] alloys. V. B. BROWNE (U.S.P. 1,570,229, 19.1.26. Appl., 19.3.25).—Iron is refined and additions of iron ore are made to the bath until no further reduction of the carbon content is obtained. Air or other oxidising gas is then caused to permeate the bath until substantially all of the remaining carbon is removed. The metal is removed from the bath and the necessary silicon addition made to adapt the silicon–iron for use as a magnetic alloy, the carbon content being not over 0·03%.

T. H. BURNHAM.

Treatment of iron or steel for preventing oxidation or rusting. T. W. Coslett (E.P. 247,071, 26.6.25).—Iron or steel surfaces are provided with a corrosion-resisting coating by subjecting them to the action of a solution such as is described in E.P. 8667 of 1906 and 28,131 of 1909 (B., 1907, 207; 1911, 92), *e.g.*, acid zinc phosphate solution, to which is added a compound of boron, *e.g.*, boric acid or borax. Before treatment the iron or steel may be immersed for a short time in a solution of boric acid. During treatment an electric current may be passed from the article as anode to a zinc or iron cathode. T. H. Burnham.

Degassing and deoxidising steel. S. Peacock and N. E. Cook (U.S.P. 1,570,176, 19.1.26. Appl., 3.3.25).—Anhydrous trisodium aluminate is added to molten iron or steel, whereby compounds with any eutectoids present are formed which melt in the bath of metal, and any compounds of oxygen and carbon associated with the eutectoids are liberated. T. H. Burnham.

Alloy steel. W. P. Woodside and C. N. Dawe, Assrs. to Studebaker Corp. (U.S.P. 1,572,458, 9.2.26. Appl., 24.8.23).—The steel contains 0·10—0·23% C, 0·30—0·60% Mn, 1·40—1·75% Ni, 0·20—0·30% Mo, and 97—98·1% Fe. T. H. Burnham.

Annealing iron articles. H. T. Grafton and C. W. Weesner (U.S.P. 1,572,600, 9.2.26. Appl., 30.3.21).—The process consists in the use of purified producer gas substantially free from oxygen and containing an insufficient amount of moisture to stain the iron articles. T. H. Burnham.

Deposition of aqueous mineral suspensions. I. Traube (E.P. 246,361, 7.5.25).—The deposition of ores from their aqueous suspensions or slimes is accelerated by the addition of an extract of a plant such as Carragheen moss or Iceland moss, with or without a small proportion of a salt or other electrolyte such as an alkaline-earth sulphate or carbonate. H. Holmes.

Reverberatory [puddling] furnaces. E. C. Lowndes (E.P. 246,681, 19.2.25).—A reverberatory furnace, particularly for puddling, is fired with pulverised fuel which is projected with a limited amount of air into a small preliminary combustion chamber where ash is deposited and the organic matter gasified; additional air enters as the gases pass over the fire-bridge into the main part of the furnace. B. M. Venables.

Nickel-copper alloy. J. W. Lehr, Assr. to U.S. Industrial Alcohol Co. (U.S.P. 1,567,066, 29.12.25. Appl., 28.8.23).—A copper–nickel alloy containing Ni 20—40% (preferably 35—40%), Sn 1—6% (2·5%), Pb 1—8% is cheaper than Monel metal while possessing all the good qualities of the latter. T. S. Wheeler.

Alloy. J. H. L. De Bats, Assr. to De Bats Metals Co. (U.S.P. 1,572,593, 9.2.26. Appl., 24.1.24).—The alloy contains 15—30% of nickel and the balance two metals of the chromium group, each not less than 15%. T. H. Burnham.

Carbonising box. C. E. MacQuigg, Assr. to Electro Metallurgical Co. (U.S.P. 1,568,174, 5.1.26. Appl., 21.11.21).—Ferrochromium alloys containing about 20% Cr and 5—7% Cu resist the action of both oxygen and carbon at high temperatures, and on this account are of value in the manufacture of boxes for use in case-hardening steel. The resistance to carbonising is due to the presence of copper, and to oxidation to the presence of chromium. T. S. Wheeler.

Treating metals. D. H. Crosser, Assr. to Chicago Crucible Co. (U.S.P. 1,568,271, 5.1.26. Appl., 29.8.24).—Reagents for treating metals are finely divided and briquetted with a large excess of clay to effect better dispersion of the reagents throughout the molten metal. For example, briquettes containing hydrated aluminium silicate (69·5%), nickel (10·4%), ferromanganese (5·6%), ferrosilicon (8·3%), ferrochromium (3·5%), and aluminium (2·7%) are added to iron in a cupola in the proportion of 1 pt. to each 100 pts. of iron. The process can also be applied to the treatment of iron in the blast furnace or the hearth, and to the treatment of copper or aluminium. T. S. Wheeler.

Alloy. P. Girin, Assr. to Soc. Anon. de Commentry, Fourchambault et Decazeville (U.S.P. 1,572,996, 16.2.26. Appl., 9.12.20).—See E.P. 159,857; B., 1922, 470 a.

Apparatus for refining or separating metals. H. Harris (U.S.P. 1,573,829, 23.2.26. Appl., 8.12.22). —See E.P. 199,660; B., 1923, 783 a.

Refining lead. H. Harris (U.S.P. 1,573,830, 23.2.26. Appl., 8.7.24. Renewed 20.10.25).—See E.P. 189,013; B., 1923, 60 a.

Cement from blast-furnace slag (G.P. 421,776). —See IX.

Zinc white (F.P. 595,396).—See XIII.

XI.—ELECTROTECHNICS.

Inductive heating. E. F. Northrup (J. Franklin Inst., 1926, 201, 221—244).—In ironless inductive heating, the principles of which are fully discussed, the necessity of making into ring form the substance to be heated is dispensed with, thus rendering the choice of shape unlimited and permitting of a furnace of crucible form with minimum radiation surface for a given volume. The inductor coil of a metal-melting furnace is a single-layer winding of flattened copper tube or solid copper strap several times as wide as it is thick and is wound edgewise. The "coupling" between the coil and the melt is chiefly determined by the ratio, square of the diameter of the melt divided by square of diameter of the coil. The frequency N is derived from the formula $N \geqq 25\times10^3/\rho D^2$, where ρ is the resistance in ohms between opposite faces of a cm. cube of the materia

and D is the diameter in cm. of the cylindrical charge to be melted. For small furnaces the high frequency needed is best obtained by the discharge-gap oscillator. It is not usual to obtain the high-frequency current by the use of oscillators when the power required for a single unit exceeds about 25 kw. The efficiency of an ironless induction furnace theoretically quite equals that of an induction furnace with transformer iron. Success in working depends on the fact that long wave-length electromagnetic radiation passes without hindrance through any heat insulator into the mass, while short wave-length radiation can only escape with difficulty from the mass, which accounts for the heat-accumulation in the charge and the attainment of very high temperatures. Molten conducting matter acted on inductively is automatically stirred and when alloys are melted the constituents mix thoroughly. Several other advantages of inductive heating are enumerated.
M. COOK.

See also A., Mar., 229, **Detection of wireless waves by crystals of galena and pyrite** (GAUBERT). 247, **Temperature coefficients of reference electrodes** (BIRCHER and HOWELL); **Measurements with quinhydrone electrode** (LINDERSTRÖM-LANG); 248, **Electrolysis of oxides dissolved in boric acid or in borates** (ANDRIEUX); **Electrolytic deposition of carbonaceous iron from complex compounds with organic acids** (SCHMIDT); **Mercury cathodes in electrolytic oxidation processes** (STSCHERBAKOV). 251, **Electrolysis of lead chloride** (SAXON). 260, **Formation of ozone from air under pressure; Ozonisation with high-frequency current** (VON WARTENBERG and TREPPENHAUER).

Lubricating oil for electricity meters. HOLDE and VON SCHACHENMEIER.—See II.

Influence of alternating currents on electrolytic corrosion of iron. ALLMAND and BARKLIE.—See X.

PATENTS.

Electrodes for electrolytic decomposition apparatus. C. F. HOLMBOE (E.P. 246,228, 29.10.24). —A plate electrode is provided with ribs on both sides of sufficient size to provide the greater portion of the active electrode surface. The arrangement and position of the ribs may be varied as desired, *e.g.*, to allow circulation of the liquid. Such electrodes, other conditions being equal, produce greater quantities of gas and require lower voltages.
J. GRANT.

Storage-battery separator. P. E. NORRIS, Assr. to WESTINGHOUSE UNION BATTERY Co. (U.S.P. 1,567,747, 29.12.25. Appl., 1.11.23).—Wood pulp, flour, or other porous material is treated with rubber latex to form a storage battery separator. The rubber latex coats the porous material without penetrating it.
T. S. WHEELER.

Voltaic cell. M. E. CONRAD, Assr. to C. B. SCHOENMEHL, INC. (U.S.P. 1,567,838, 29.12.25. Appl., 9.2.21).—Cells of the type described in U.S.P. 719,752 (B., 1903, 304) have their resistance decreased and voltage increased by forming the negative electrode of copper oxide or other suitable metal oxide which has been treated with the fumes or vapour of sulphur at a temperature above the boiling point of sulphur.
T. S. WHEELER.

Carbon electrodes for primary cells using air-depolarisation. COMP. LORRAINE DE CHARBONS, LAMPES ET APPAREILLAGES ÉLECT. (F.P. 594,697, 31.5.24).—The electrodes consist of active carbon, graphite, or retort carbon, and an oxidising catalyst, *e.g.*, a compound of manganese, copper, silver, vanadium, uranium, titanium, iron, or cobalt.
J. GRANT.

Electrolysis of alkali chlorides. F. GERLACH (F.P. 596,918, 20.4.25. Conv., 21.5.24).—In the electrolysis of alkali chlorides with mercury cathodes, the mercury liberated by decomposition of the alkali amalgam is led to the amalgam-formation chamber in presence of liquid (water, alkali chloride solution, dilute alkali lye) and, if desired, in an atmosphere of an inert gas.

Manufacture of electrode-carbon. C. EHRENBERG, H. WIEDERHOLD, C. KRUG, M. G. HOLSBOER, K. FISCHER, and STUDIENGES. FÜR AUSBAU DER IND. (G.P. 422,235, 30.11.23).—Humic acids are liberated by acid from the solution produced by treatment of substances containing humic acid (*e.g.*, humic coal, peat, etc.) with alkali, and are then carbonised.
J. GRANT.

Material for self-burning electrodes. NORSKE A./S. FOR ELEKTROCHEM. IND. NORSKE IND.-HYPOTEKBANK (Swiss P. 111,653, 23.7.24. Conv., 25.7.23).—The electrodes consist of metal casings containing a mixture of carbon particles of varying degrees of subdivision, together with so much (more than 18%) binding material (tar and pitch) that the mass when warm is no longer capable of being compressed by stamping. During heating this filling coheres to form a mass having a high density.
J. GRANT.

Electrical gas purification. SIEMENS-SCHUCKERTWERKE GES.M.B.H. (G.P. 422,619, 28.2.24).—The gas is led first in contact with precipitating electrodes arranged along the direction of gas flow, and then through permeable electrodes placed across the flow.
E. S. KREIS.

Manufacture of electroionic discharge tubes. EDISON SWAN ELECTRIC CO., LTD., Assees. of RADIO-RÖHREN-LAB. G. NICKEL GES.M.B.H. (E.P. 232,978, 24.4.25. Conv., 28.4.24).—In a thermionic discharge tube, the effects of space charge are limited by using an incandescent cathode (and/or an auxiliary heated electrode) which is covered with a chemical compound (*e.g.*, calcium hydride) which is highly emissive of electrons and which disintegrates gradually producing positive ions. The tube is either wholly evacuated or contains no gas other than that given off by the heated compound. Other substances either for binding and/or for the production of electrons and positive ions

may be mixed with the disintegrating compound. The grid of ions which is formed in close proximity to the incandescent filament has practically the same properties as an electrically charged stable grid. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 230,226.) A. W. Hothersall.

Deposition of chemical compounds on incandescence bodies. N.V. Philips' Gloeilampenfabr. (F.P. 594,165, 8.10.24. Conv., 11.10.23).—Solid chemical compounds (nitride and phosphide of zirconium, carbides of molybdenum and tantalum, etc.) are produced by passing the vapours from certain of their volatile compounds, usually mixed with hydrogen or nitrogen, over a metal filament heated to 1000—1800°. J. Grant.

Impregnated insulation. G. F. Dreher, Assr. to Gen. Electric Co. (U.S.P. 1,574,562, 23.2.26. Appl., 9.6.23).—See E.P. 228,224; B., 1925, 249.

See also pages 256, **Carrying out and controlling reactions** (G.P. 422,159). 261, **Dust removal in briquetting works** (G.P. 422,148). 262, **Separating condensable products from gases** (G.P. 422,121). 274, **Detecting oxygen** (U.S.P. 1,567,534). 283, **Treating iron or steel to prevent corrosion** (E.P. 247,071). 289, **Manufacture of rubber goods** (E.P. 246,532). 297, **Electrical apparatus for preserving fodder** (G.P. 418,481). 299, **Lead tetraethyl** (U.S.P. 1,567,159).

XII.—FATS; OILS; WAXES.

Separation of ricinoleic acid from the mixed fatty acids of castor oil. K. Inokuchi (J. Soc. Chem. Ind. Japan, 1925, **28**, 1353—1358).—The solubilities of barium ricinoleate and barium stearate in absolute alcohol (g. per 100 g. of solution) are: 10°, 0·06 g., trace; 20°, 0·13 g., trace; 30°, 0·325 g., trace; 40°, 2·00 g., 0·014 g.; 50°, 9·372 g., 0·018 g.; 60°, 28·19 g., 0·024 g. In alcohol of d_4^{15} 0·8119 the solubility of barium stearate is trace at 20°, 0·011 g. at 30°, 0·017 g. at 40°, 0·022 g. at 50° and 0·029 g. at 60°. For preparing pure ricinoleic acid from castor oil fatty acids by the barium salt–alcohol process, it is recommended that the temperature be 50°, the time for dissolving the mixed barium salts in the solvent about 1 hr., that as solvent first 95% (by weight) alcohol, then 98%, and finally absolute alcohol be used, and that the amount of alcohol for one separation be about 100 c.c. for each 10 g. of the salts. K. Kashima.

Highly unsaturated acids in ox liver oil. K. Kimura (J. Soc. Chem. Ind. Japan, 1925, **28**, 1366—1373).—An oily liquid obtained by direct heating of ox liver in a pan was extracted with ether and the extract was treated with acetone and alcohol. Ox liver oil (soluble in ether and acetone) thus obtained had the following characters: d_4^{100} 0·9008, acid value 36·5, saponif. value 128·3, iodine value 113·3, n_D^{60} 1·4720, and unsaponifiable matter 40·62%. The fatty acids freed from unsaponifiable matter had m.p. 39—42°, d_4^{100} 0·8437, neutralisation value 196·3, iodine value 104·4, n_D^{60} 1·4480, ether-insoluble bromides 19·2%. The corresponding values for the fatty acids from the portion insoluble in acetone but soluble in alcohol and from the portion insoluble in both alchool and acetone were, respectively: m.p. 48·5—51°, 51—54·5°, d_4^{100} 0·8515, 0·8521, neutralisation value 188·6, 186·0, iodine value 132·1, 143·5, n^{60} 1·4522, 1·4529, ether-insoluble bromides 30·0%, 38·3%. The ether-insoluble bromides had nearly the same composition in all cases. The bromides were debrominated and the highly unsaturated acids thus obtained were converted into methyl esters and subjected to distillation. Hydrogenated products of some fractions contained arachidic and behenic acids. The presence of $C_{20}H_{32}O_2$ (cf. Hartley, J. Physiology, 1907, 17; 1909, 353) and of $C_{22}H_{36}O_2$ in the oil was proved. Acids of the $C_nH_{2n-10}O_2$ series are either absent or present only in minute amount. The odour of the highly unsaturated acids from ox liver oil resembles that of the acids from fish oil. K. Kashima.

Vitamin potency of cod-liver oils. XVIII. Effect on vitamin potency of cold pressing cod-liver oils. A. D. Holmes and M. G. Pigott (Ind. Eng. Chem., 1926, 18, 188—189).—The stearine solidified by refrigeration in a brine-cooled tank was removed from the liquid oil. Chemical analysis of the crude and pressed oils revealed little difference between them, and tests on the vitamin potency, although not showing identical results, did not indicate any significant divergencies. D. G. Hewer.

Examination of cacao-fat for alkalis and alkaline-earths. J. Prescher and R. Claus (Z. Unters. Nahr. Genussm., 1925, **50**, 429—430).—The occurrence and determination of alkalis or alkaline-earths in animal and cacao fats are discussed, and the official method of analysis for the former case is applied to the latter. This involves the precipitation of the acidified water-extract of the fat with ammonia and ammonium carbonate solutions. The natural lime-content of cacao-fat (about 0·01%) is greater than that of animal fat. J. Grant.

Use of sintered glass crucibles for the separation of digitoninsteride in the examination of fats for phytosterol. J. Prescher and R. Claus (Z. Unters. Nahr. Genussm., 1925, **50**, 420—423).—Jena sintered glass crucibles and filter-flasks have been found to be highly satisfactory for the separation of the digitonin precipitate in the isolation of sterols (phytosterol, cholesterol) from fats, prior to conversion into the actates for identification purposes. J. Grant.

Detection and determination of small amounts of chromium in fats. A. O. Snoddy (J. Oil and Fat Ind., 1925, **2**, 20—25).—200 g. of fat are ignited in a muffle, the ash is fused with sodium and potassium carbonates, and the aqueous solution of the fused mass, after acidification with hydrochloric acid, is diluted to 200 c.c. An aliquot part of this

solution, after addition of 5 c.c. of 20% hydrochloric acid and 1 c.c. of diphenylcarbazide solution (0·1—0·2 g. in 10 c.c. of glacial acetic acid, diluted to 100 c.c. with 95% alcohol), is compared colorimetrically with potassium dichromate and carbonate solution similarly treated, the violet colour reaching a maximum intensity within 10 min. Small amounts of iron, aluminium, copper, and silicon do not interfere. A. A. ELDRIDGE.

Autoclave fat-splitting. H. RIEMER (Z. Deuts. Oel- u. Fett-Ind., 1925, 45, 653—655, 701—703, 720—722, 736—739; Chem. Zentr., 1926, I, 1323—1324).—The author gives a brief characterisation of the evolution of industrial fat-splitting as well as the theoretical explanation thereof due to Harkins and Langmuir. The suitability of different metals for autoclave construction is discussed, cast-iron not being recommended for high-pressure work. An aluminium alloy is found especially suitable as it is not affected by fatty acids or condensed water, is not attacked by zinc oxide or magnesia, and is suitable mechanically. A detailed description of the author's high-pressure process (cf. Austr. P. 101,041; B., 1926, 200) is given, the following advantages being claimed :—Pressures of 18—25 atm. may be used; there is no anhydride formation; no previous purification of the fat is necessary; deodorisation is effected, so that there is the possibility of treating all kinds of raw materials, especially marine animal oils. H. M. LANGTON.

Catalytic hydrogenation of highly unsaturated acids. I. Course of hydrogenation of methyl esters of highly unsaturated acids in the presence of nickel catalyst. Y. TOYAMA and T. TSUCHIYA (J. Soc. Chem. Ind. Japan, 1925, 28, 1079—1087).—The methyl esters of the highly unsaturated acids obtained from Japanese sardine oil have been hydrogenated in the presence of nickel at 185—210° under ordinary pressure. The mixed esters had b.p. 210—227°/5 mm., saponif. value 169·2, iodine value 350·6; the fatty acids obtained from the esters yielded 127·1% of ether-insoluble bromide containing 71·06% Br. The individual acids which may possibly have been present in the original sample are $C_{20}H_{30}O_2$, $C_{22}H_{34}O_2$, $C_{20}H_{32}O_2$, and $C_{22}H_{36}O_2$. Before the iodine value of the original sample fell to 11·4, portions were taken out at 12 different stages of hydrogenation and analysed. The content of fatty acids giving ether-insoluble bromide steadily decreased as hydrogenation proceeded. Thus when the iodine value fell to 173·1 the amount of ether-insoluble bromide was 1·67%, and when the iodine value was 134·9 no bromide was produced. As the bromide content of ether-insoluble bromide was more than 69% in every case, it may be concluded that no substantial amount of fatty acids giving ether-insoluble hexabromide was formed during hydrogenation. The fact that the bromine content of the ether-insoluble bromide decreased somewhat with the progress of the hydrogenation may be accounted for by partial conversion of the fatty acids with five double bonds into those with four double bonds. Until nearly all of the fatty acids giving ether-insoluble bromide disappeared no appreciable amount of solid acid was formed, but immediately after this stage solid acids were formed in substantial amount. It is probable, therefore, that until nearly all of the fatty acids giving ether-insoluble bromide have disappeared, the hydrogenation results mainly in the conversion of highly unsaturated acids into acids with two double bonds, and that subsequently the chief change is the conversion of the acids with two double bonds into those with one double bond, with formation also of saturated acids. Even in the sample having an iodine value of 134·9, the presence of saturated acids is indicated. The solid acids with one double bond formed during the hydrogenation include acids having markedly higher m.p. than naturally occurring C_{20} and C_{22} acids of the oleic series, and it is even probable that solid acids with two double bonds are formed.
K. KASHIMA.

Naphthenic soap. J. MIKUMO (J. Soc. Chem. Ind. Japan, 1925, 28, 1121—1126).—Sodium soap, prepared from a mixture of Japanese naphthenic acids (d_4^{15} 0·9708, acid value 249·2), was examined with regard to its surface and interfacial tensions, and emulsifying, lathering, and detergent powers. Parallel tests were made with sodium soaps from Kahlbaum's oleic acid, coconut oil, and rosin. The naphthenic soap is extremely soluble in cold water, and is superior even to coconut oil soap in lathering power. It is, however, soft and is far inferior to the oleate in detergent action. It may be regarded as intermediate between fatty and rosin soaps.
K. KASHIMA.

Determining palm kernel oil and butter in margarine. ELSDON and SMITH.—See XIX.

PATENTS.

Preparation of emulsifying agents from castor oil. J. B. G. AUBRY (F.P. 592,764, 30.1.25).—Castor oil is polymerised by heating in an inert atmosphere at 240°, the product being sulphonated, allowed to remain for 48 hrs., washed with water containing formaldehyde, and then neutralised by addition of ammonia. A. J. HALL.

Preparation of mixed esters of fatty acids. OELWERKE GERMANIA G.M.B.H., and W. NORMANN (G.P. 417,215, 26.6.20).—Mixtures of esters of fatty acids are heated at a high temperature for a prolonged period, with or without a catalyst, whereby mixed esters having a lower melting point are formed. Products closely resembling butter fat may be thus obtained. Non-esterified products may be removed from the resulting esters by special methods, *e.g.*, distillation. Suitable catalysts include aromatic and aromatic–aliphatic sulphonic acids, tin and its salts, and alkali alkoxides. For example, a mixture of 10% of tristearin and 90% of soya bean oil having m.p. 54·5° yields when heated at 250° for 9·5, 17, and 24 hrs. products having m.p. 50·8°, 43·7°, and 34·3° respectively. A mixture of 2 mols.

of tristearin and 1 mol. of triacetin is hard, brittle, and friable but on heating for 72 hrs. at 200—250° it loses its brittleness and becomes plastic. A mixture of equal parts of neutral coconut oil and ethyl stearate heated for 18 hrs. at 250° in the presence of 1% of tin and in a stream of carbon dioxide (for removal of volatile products) rapidly yields the ethyl esters of lower fatty acids having an easily recognised odour; the distillate has a saponification value of 221, whilst the saponification values of ethyl stearate and the ethyl esters of coconut oil acids (mean mol. wt., 202) are 180 and 244 respectively. When a mixture of 20 pts. of tristearin, 80 pts. of coconut oil, and 1% of naphthalenesulphonic acid is heated for 2·5 hrs. at 250°, the product has m.p. 34·5° (m.p. of original mixture is 57·3°); when $\frac{1}{2}$% of sodium ethoxide is used as a catalyst and the heating is carried out for $\frac{1}{2}$ hr. *in vacuo* at 140°, the product has m.p. 31·4°. A. J. Hall.

Extraction of mixtures of oils and fats by means of alcohol. K. Kubierschky (G.P. 417,964, 28.5.20).—The alcoholic extract is treated with sufficient water so that a part of the alcohol-soluble substances is precipitated together with any dissolved neutral oil; a more valuable extract and a higher yield of neutral oil are thereby obtained.

A. J. Hall.

Preparation of fats from fatty acids with an odd number of carbon atoms. Farbenfabr. vorm. F. Bayer u. Co., Assees. of S. Dreichsel and H. Weyland (G.P. 422,687, 12.4.24).—A fatty acid mixture consisting mainly of lauric acid is converted into the corresponding methyl ketones, the latter are oxidised, and the resulting fatty acids are converted into glycol or glycerol esters in the usual manner. Tasteless and odourless, readily assimilable fats are obtained.

H. M. Langton.

Hydrolysing fats, oils, and waxes. W. Schrauth (Can. P. 245,373, 10.3.24).—Fatty substances are heated with aqueous solutions of sulphonic acids of polynuclear hydrocarbons substituted with alkyl groups, *e.g.*, *iso*propyl, in the nucleus.

A. J. Hall.

Chromium soap. T. T. Gray (U.S.P. 1,567,049, 29.12.25. Appl., 3.10.22).—A mixture of an alkali soap, such as sodium stearate, with 1/5th to 5 times its weight of a chromium soap, such as chromium stearate, is non-irritant, has good lathering and detergent properties, no objectionable taste, and is particularly suitable for use as a shaving soap or as a dentrifice. The chromium stearate is prepared by the interaction of chromium hydroxide and molten stearic acid or by saponifying a fatty acid or a fat with a mixture of sodium and chromium hydroxides.

T. S. Wheeler.

Obtaining sulphonated products of wool fat. O. Herzog (E.P. 247,714, 10.1.25).—See U.S.P. 1,543 157 ; B., 1925, 680.

Acetylenation of fatty substances (U.S.P. 1,567,785).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Barium sulphate [heavy spar and *blanc fixe*]. C. P. van Hoek (Farben-Ztg., 1926, 31, 1136—1137).—The undesirable properties conferred on a paint by the presence of barium sulphate in the form of heavy spar or *blanc fixe* finds a parallel in rubber mixes. The presence of an adsorbed layer of air on the particles inhibits adequate adhesion to the oil medium in paints and is suggested as being the cause of the low opacity and the weakening of paint films. A. de Waele.

Influence of number and size of particles on the covering power [of pigments]. C. Kühn (Farben-Ztg., 1926, 31, 1131—1133).—The relative opacities of unit volumes of suspensions of burnt sienna in boiled linseed oil were determined by viewing under a low-power microscope illuminated by diffused candle light, and noting the opacity values of smoke-glass oculars necessary to obtain complete extinction of the light transmitted. It was found that the opacities were proportional to the number of particles per unit volume of suspension, further tests confirming the fact that the relative dimensions of the particles (between the limits of 159 and 283 cm.^{2} specific surface examined) did not affect the opacities. The relationship between specific surface and opacity is similarly linear, but the rate of increase of opacity with increase in the number of particles per unit volume of suspension increases more rapidly in the case of the finer particles (cf. B., 1920, 697 A). A. de Waele.

Settling and packing of mixed paints. W. C. Arsem (Ind. Eng. Chem., 1926, 18, 157—160).—The tendency of the particles of paint pigments to flocculate and of driers and soaps in the vehicle to gelatinise is inhibited by the presence of free acid or resinous dispersing agents. On storage, a slow combination of basic pigments with the peptising agent may occur, prejudicing the stability of the vehicle sol and producing basic soaps which have very little dispersing action on a pigment derived from the same metal, thus permitting flocculation and caking. The alteration in acidity and ash of the medium in stored zinc oxide paints was observed over a period of 22 months, and the ratio of ash to total acid (calculated as oleic acid) approached the constant value required by the formation of basic zinc oleate of known constitution.

S. S. Woolf.

Factors determining the brightness and opacity of white paints. F. H. Rhodes and J. S. Fonda (Ind. Eng. Chem., 1926, 18, 130—135). The hiding power and whiteness of a film of white paint is dependent on thickness of film, refractive index of pigment and of medium, particle size of pigment, and percentage volume of pigment in the paint. Earlier determinations of opacity using films of liquid paint are adversely criticised. In the present investigation dried films of various thicknesses over black were examined for brightness by an integrating photometer (cf. Taylor, U.S. Bur. Standards, Sci.

Paper 405). The conception of " ultimate brightness" —not increased by further increase in the thickness of the film—is introduced, and a formula correlating the above factors is developed and receives support from the experimental results. An explanation is offered of the increase in opacity obtained by adding small quantities of dark pigments to a white paint.
S. S. WOOLF.

Evolution of vapours containing nitric oxide, carbon monoxide, and hydrogen cyanide in the decomposition of triolin. E. WILKE-DÖRFURT, A. SIMON, and E. GÜHRING (Z. angew. Chem., 1926, **39**, 196—198).—Triolin, a new form of flooring material (cf. Simon, B., 1926, 21), decomposes at about 125° with the formation of highly poisonous vapours containing 8—10% HCN and considerable proportions of nitric oxide and carbon monoxide. The hydrogen cyanide is formed by the decomposition of the triolin itself, and by a secondary reaction between carbonaceous matter and the nitric oxide first formed, as well as by the decomposition of the Prussian blue used for colouring purposes. Even at room temperatures appreciable quantities of nitric oxide are formed in a current of air passing over the triolin.
B. W. CLARKE.

See also A., Mar., 243, **Swelling and dispersion of colloidal substances in ether-alcohol mixtures.** (MARDLES). 259, **Ferro- and ferricyanides** (TARUGI).

PATENTS.

Zinc white. R. D. LANCE (F.P. 595,396, 17.6.24). —The mixture of carbon monoxide and zinc vapour, resulting from heating oxide or sulphide ores with coal, is burnt in the neck of the muffle or at the gas outlet by blowing in air. Combustion of the carbon monoxide assists that of the zinc, and the product is free from metal.
A. GEAKE.

Acetylenation of fatty and other substances. [Preparation of resinous substances.] L. G. BOURGOIN (U.S.P. 1,567,785, 29.12.25. Appl., 23.10.24).—The fat or other substance under treatment is mixed with a molecular proportion of calcium carbide and heated with a salt containing water of crystallisation or treated under pressure with steam at a temperature above the melting point of the substance, *e.g.*, 100—350°. The products are plastics and resins suitable for use as insulators, artificial rubber, or the like. For example, 5—8 pts. of carbide are mixed with 100 pts. of an animal or vegetable fat or the corresponding amount of glycerin and fatty acid and treated with steam under pressure at 180—250° for 2 hrs. According to the conditions of treatment there is obtained a more or less plastic and malleable substance which is easily moulded when hot and is insoluble in water, acetone, alcohol, and ammonia but soluble in carbon tetrachloride, carbon disulphide, and ether.
T. S. WHEELER.

Purification of spirit-soluble Manila copal. L. BLUMER CHEM. FABR. (G.P. 420,918, 29.7.24).—The pulverised copal is refluxed with limited quantities of solvent until it is well swollen, whereupon the solvent is distilled off, the temperature raised to about 200°, and finally the mobile liquid mass is filtered. The copal may be mixed with other resins.
E. H. SHARPLES.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

***Hevea* latex. IV. Proteins. V. Possible occurrence of a coalescing enzyme.** W. N. C. BELGRAVE (Malay. Agric. J., 1925, **8**, 367—375 ; cf. B., 1925, 771).—By the successive addition of alcohol and sodium hydroxide solution to diluted *Hevea* latex coagulation is effected, but the serum retains a considerable proportion of the protein which can be precipitated by regulated acidification. Analysis of the protein reveals a fairly uniform distribution of nitrogen between the diamino-, monoamino-, and non-amino-groups. The views of de Vries (B., 1924, 797, 798; 1925, 17) as to the existence of a coalescing enzyme in latex are rejected. Protein substances rather than resins are probably associated with the coagulation phenomena, but the opposing views could be reconciled by the occurrence of a lipin–protein complex unstable towards heat.
D. F. TWISS.

Rubber coagulated with sodium silicofluoride. J. EDWARDES (Bull. Rubber Growers' Assoc., 1926, **8**, 34—36).—The use of sodium silicofluoride as coagulant in conjunction with that of *p*-nitrophenol as mould-preventive, prevents the development of the violet colour frequently observed in rubber prepared with the aid of the latter substance and acetic acid ; it also renders unnecessary the addition of sodium bisulphite to latex for the production of pale crêpe rubber, and checks the tendency to bubble formation in smoked sheet rubber.
D. F. TWISS.

Coagulation and mould prevention of smoked sheet rubber. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1926, **8**, 36—40; cf. preceding abstract).—Rubber prepared with the aid of *p*-nitrophenol vulcanises rather more rapidly than the standard or than rubber prepared with dinitrophenol; the retarding effect of sodium silicofluoride is therefore compensated to some extent by the simultaneous use of *p*-nitrophenol. Sodium silicofluoride prevents bubbles in smoked sheet rubber.
D. F. TWISS.

Alteration of the degree of dispersion of rubber. F. EVERS (Kautschuk, 1925, Nov., 8—9).— If rubber is treated in boiling chlorobenzene or xylene solution with aluminium chloride, the resulting solution on addition of alcohol yields a precipitate which gradually hardens. The product gives a benzene solution of very low viscosity and evidently represents a much lower state of " aggregation " than that of the original rubber.
D. F. TWISS.

Influence of state of subdivision [of rubber] on the period necessary for extraction with acetone [to determine resin]. S. REINER (Kautschuk, 1926, Jan., 5—6).—Experiments with

an apparatus especially designed to give uniform results show that the size of the fragments of Para rubber greatly influences the rate of extraction of the resins by acetone. The results indicate that the customary period of extraction is excessive.
D. F. Twiss.

Chemistry of rubber and gutta-percha. Isoprene and caoutchouc. X. H. Staudinger (Kautschuk, 1925, Aug., 5—9; Sep., 8—10).—Rubber, polysaccharides, proteins, and many synthetic products are "eucolloids" in which the primary colloidal particles are identical with the molecules (macromolecules), whereas with pseudocolloids, such as salts of the fatty acids, the primary colloidal particles result by association of the molecules. Association involves forces similar to those active in crystallisation, whereas in polymerisation the fundamental molecules undergo chemical change. Styrene is a "hemicolloid" containing both molecularly complex eucolloid material and also substances of low molecular weight. Hemicolloids are also obtained in the degradation of rubber. The chemical nature of *cyclo*caoutchouc, hydrocaoutchouc, rubber (natural and synthetic), and gutta-percha is discussed briefly as also is the chemical effect of heat on rubber.
D. F. Twiss.

Effect of accelerators on cure [vulcanisation] and quality of various rubbers. R. P. Dinsmore and A. O. Zimmerman (Ind. Eng. Chem., 1926, **18**, 144—157).—The two most significant properties for the comparison of various rubbers are the "stiffness" and the "tear resistance"; the "best cure" of rubber is the period of vulcanisation at a given temperature at which these properties approach the most satisfactory balance with respect to each other and with respect to their values after ageing. Slope, tensile strength, ultimate elongation, tensile product, hysteresis, permanent set, energy of resilience, and coefficient of vulcanisation are all less satisfactory as criteria of the quality of rubber and of its variation with vulcanisation. Although the difference in rate of vulcanisation may be reduced by the introduction of an accelerator into a rubber–sulphur mixture, accelerated mixings are not necessarily more uniform and two rubbers with the same rate of vulcanisation in simple admixture with sulphur may exhibit different rates of vulcanisation with the addition of an accelerator. It is therefore insufficient to use one or even two or three mixings to evaluate rubber for use with various accelerators. Pale crêpe, on the whole, is more uniform than smoked sheet rubber, but unfortunately its quality is uniformly on the low side. The mixing used for the tests was rubber (100), sulphur (variable), accelerator (variable), and zinc oxide (6). By "stiffness" is meant the increase in load necessary to increase the elongation from 600 to 800% for an unaccelerated mixing or from 500 to 700% if accelerated.
D. F. Twiss.

Estimation of fineness of mineral fillers in the rubber industry. F. Kirchhof (Kautschuk, 1925, Sep., 17—18).—A convenient rough method for estimating the relative particle size of various powders intended as rubber ingredients is to place from 5 to 20 g. in a graduated 100 c.c. tube 25 cm. long and to tap until the bulk becomes constant; the calculated final volume per gram (or per gram/specific gravity) forms a comparative index of the fineness.
D. F. Twiss.

Heavy spar and *blanc fixe*. Van Hoek.—See XIII.

Patents.

Preserving vulcanised rubber. Robel und Fiedler G.m.b.H. (E.P. 221,788, 26.8.24. Conv., 13.9.23).—The deterioration of vulcanised rubber on storage is counteracted by treatment with a mixture of glycerol and benzaldehyde. A suitable solution is prepared by adding a mixture of glycerol (172 g.), benzaldehyde (34 g.), and talcum (1300 g.) to a hot solution of rubber (60 g.) in paraffin oil (600 g.) which has been heated for 4 hrs. at 140°; phenylhydrazine (28 g.) or glucose (20 g.) may also be introduced; the intimate mixture is then stirred at 180° for 2 hrs. The cold mixture is applied with a hard brush.
D. F. Twiss.

Manufacture of rubber goods from rubber emulsions. Anode Rubber Co. From P. Klein and A. Szegvári (E.P. 246,532, 28.7.24. Addn. to 223,189; B., 1925, 46).—In addition to the protection of the rubber deposit from contact with the anode gases by mechanical means, the production of homogeneous electrophoretic deposits of rubber can be ensured by prevention of the liberation of gases. The addition of sodium chloride and pyrogallol to preserved latex, or the impregnation of a porous mould, surrounding the anode, with manganous oxide prevents the liberation of gaseous oxygen. Alternatively a similar end may be achieved by using a zinc anode with latex containing sodium chloride. By using a carbon anode with latex to which sodium thiosulphate has been added, sulphur is deposited together with the rubber. The thickness of the rubber deposit is influenced by the material of the mould; the deposit forms more rapidly on gypsum than on unglazed earthenware. Fabric-lined rubber goods can be produced by slipping over an anode-mould of the desired shape the necessary fabric lining and depositing the rubber thereon.
D. F. Twiss.

Hard resinous vulcanisation accelerator. W. Scott, Assr. to E. I. du Pont de Nemours and Co. (U.S.P. 1,571,739, 2.2.26. Appl., 19.12.22).—An accelerator for the vulcanisation of rubber is formed by the action of formaldehyde on the condensation product of acetaldehyde (3 mols.) and aniline (2 mols.).
D. F. Twiss.

Acceleration of vulcanisation of rubber. Ricard, Allenet et Cie. (Addition 29,785, 31.5.24, to F.P. 523,417; cf. E.P. 157,050; B., 1921, 520 a).—Rubber is mixed with 0·2—1% of furfuramide or other furfuraldehyde derivatives containing nitrogen, and vulcanised at 5—6 atm. for 9—18 min.
E. H. Sharples.

Recovery of rubber and fabric from old rubber. A. L. Marchal (F.P. 593,602, 26.11.24).—Rubber goods containing fabric, such as tyres, are heated in a closed vessel at 180—200° with copper turnings and a rubber solvent; the rubber can then be removed from the adhering fabric by washing with benzine. The fabric so obtained can be used again after bleaching and the rubber is to a great extent desulphurised. E. H. Sharples.

Bituminous compositions (E.P. 246,548).—See IX.

Storage battery separator (U.S.P. 1,567,747).—See XI.

XV.—LEATHER; GLUE.

Activities of pancreatic enzymes used in bating upon different substrates. J. A. Wilson and H. B. Merrill (J. Amer. Leather Chem. Assoc., 1926, **21**, 50—53).—The activities of nine commercial pancreatins have been compared by seven methods, viz., the Fuld-Gross casein method, the methods previously quoted (cf. B., 1926, 205), and the Wohlgemuth fat method. In the Fuld-Gross method the activity equals the reciprocal of the smallest number of grams per litre of enzyme capable of digesting all the casein in a solution containing 1 g. per litre in 1 hr. at 40°. The Wohlgemuth method defines the activity as the c.c. of 0·1 N-sodium hydroxide required to neutralise the free fatty acids liberated from 10 g. of emulsified olive oil by 0·10 g. of the enzyme in 1 hr. at 40°. The Willstätter fat figure is preferred because it provides for a better control of p_H value and gives results more easily reproducible. The results show that the pancreatins used in bating possess fat-splitting powers, but these are not reliable as a measure of the activities on the skin fats. The commercial products are very variable in composition. The activities as determined by the casein method are not the same as those on collagen, elastin, and keratose. The latter should be used as substrates and not casein. D. Woodroffe.

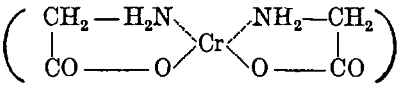
Important rôle played by enzymes in bating. J. A. Wilson and H. B. Merrill (Ind. Eng. Chem., 1926, **18**, 185—188).—When skins are limed before tanning, the keratins are partially hydrolysed into keratose, which is soluble in neutral or alkaline solution, but is precipitated at its isoelectric point, p_H 4·1. If this material is left in the skin and precipitated there by the acid tan liquors, the value of the finished leather is impaired. In bating, keratose is hydrolysed by the enzymes to such an extent that it is no longer precipitable by acid. Pancreatin hydrolyses keratose in the p_H range 5·5—11·2 only, and maximum hydrolysis is obtained at p_H 7·9. The hydrolysis at 40° and p_H 7·9 has been measured as a function of the time and concentration of the enzyme and keratose. The proteolytic powers of different enzyme preparations have been measured by their keratose-digesting power (cf. B., 1926, 205). D. Woodroffe.

Ultrafiltration of vegetable tanning solutions. A. W. Thomas and M. W. Kelly (Ind. Eng. Chem., 1926, **18**, 136—138).—The ultrafiltration of solutions of hemlock bark extract containing 0·4 g. of tannin per 100 c.c. and varying in p_H from 2·0 to 9·0 was carried out in a Columbia type of ultrafilter using compressed nitrogen at pressures of 80, 120, and 200 lb./in.^{2} The membranes were prepared by soaking filter paper in collodion solution and allowing to become "tacky" before immersion in water, according to the method of R. J. Browne (B., 1923, 989 A). Pronounced differences were observed in the amount of total solids held back both as a function of the p_H of the solutions and also, with identical solutions, as a result of differences in the collodion solutions in which the membranes were prepared and individual variations in the membranes. Variations in the gelatin-salt test for tannins in the ultrafiltrate give an additional indication of the unreliability of the method for separating tannins and non-tannins. A. W. Hothersall.

Aldehyde tannage. A. W. Thomas, M. W. Kelly, and S. B. Foster (J. Amer. Leather Chem. Assoc., 1926, **21**, 57—76).—Experiments with powdered gelatin or hide powder and formaldehyde solutions show that the tanning action is best between p_H 6·0 and 9·0. The amount of formaldehyde fixed from increasingly concentrated solutions increases but not in direct proportion. High concentrations of sodium chloride increase the rate of fixation of the aldehyde. Neutral or faintly alkaline solutions of formaldehyde are most favourable for tanning. Owing to the hydrolysis of hide powder, formaldehyde tanning tests should not be made on this material. D. Woodroffe.

Internal complex salt formation as mechanism of chrome tanning. K. H. Gustavson (J. Amer. Leather Chem. Assoc., 1926, **21**, 22—30; cf. B., 1924, 65; 1925, 604, 817).—With decrease in the percentage acidity of chrome liquors, there is an increased fixation of chromium by formaldehyde–collagen and for a moderately acid salt this increase is accentuated by increased concentration of the liquor. The grass-green colour of the tanned formaldehyde compound seems to show that the colour of ordinary chrome-tanned leather is due to internal salt formation between the secondary valencies of the chromium salts and the basic groups of the protein. Fixation of chromium-containing anions is decreased by pre-treatment of the collagen with formaldehyde. Hide powder tanned with sumach, quebracho, and tannic acid at p_H 2·0—6·0 shows decreased fixation of chromium from chromium chlorides and sulphates (up to 50% reduction). In concentrated basic liquors part of the tannin is replaced by chromium, probably due to the fixation of electro-negative sulphato-complexes. The oxalato-compound showed a proportionately lesser retardation in the chromium fixation by the tannin–collagen compounds than the salts in which chromium is present as cation. The degree of chromium fixation by hide substance from ordinary chrome liquors is

determined not only by the state of the acidic groups, but is also a function of the basic groups of the protein. Hide powder tanned by means of chromium cations shows no decreased affinity for vegetable tannins. Hide powder tanned with oxalato-chromium compounds shows a slight decrease in the rate of vegetable tanning. The reactivity of deaminised collagen and tannin–collagen compounds towards cathodic and anodic chromium is similar and this resemblance supports the inner complex hypothesis of chrome tanning, according to which the chrome–collagen compound is an internal complex salt of the type



Primary valency is probably concerned in the reaction of chromium salts with the acidic groups in collagen and secondary valency with the basic groups. Anodic chromium fixation from the oxalato-compound shows a maximum rate in the isoelectric zone of the proteins and is probably of the residual valency type. This reaction is distinctly different from the combination of chromium cations with collagen. D. WOODROFFE.

Colloid chemistry in tanning. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1926, 21, 53—57).—A reply to the criticisms of W. D. Bancroft (*ibid.*, 1925, 565) who doubts the formation of definite chemical compounds in chrome tanning (cf. B., 1925, 604). D. WOODROFFE.

Behaviour of deaminised collagen. Further evidence in favour of the chemical nature of tanning. A. W. THOMAS and S. B. FOSTER (J. Amer. Chem. Soc., 1926, 48, 489—501).—Hide powder (100 g.) was suspended in water (1 litre), and a solution of sodium nitrite (100 g.) in water (500 c.c.) added, and then glacial acetic acid (70 c.c.). After keeping for 24 hrs. the resulting canary-yellow product was collected, washed with water, and covered with sodium chloride to reduce swelling and force out more of the acid solution, salt and acid then being removed by washing with water, the product dehydrated with 95% alcohol, and dried in a current of air (cf. Hitchcock, A., 1923, i, 1244). The material thus obtained contained 0·49% less nitrogen than the original hide powder, and its isoelectric point was p_H 3·7—4·2. The p_H at which fixation of tannin by the deaminised material was a minimum was 3·0—3·5, as compared with 4—5 for the original hide powder, and, at p_H values below 4·0, the fixation of tannin by the deaminised material was less than that of the original. On the alkaline side of p_H 3·5, the rate of fixation by the deaminised material increased rapidly to a maximum at p_H 7—8, then fell to p_H 9—10. These results are in accordance with the view that tanning is a process of chemical combination between the tannin and the skin. Calf-skin, deaminised analogously, shows two points of minimum plumping, at p_H 4·0 and 8·3, and swells, in solutions of p_H 6 to 10, to a considerably greater degree than the raw skin. F. G. WILLSON.

Films of adhesives. J. W. McBAIN and D. G. HOPKINS (J. Physical Chem., 1926, 30, 114—125: cf. B., 1925, 292).—Determinations of the tensile strengths of various adhesives have been made by a method which measures the strengths of thin films prepared by allowing them to dry on a surface (a ferrotype plate coated with a trace of vaseline) to which they do not adhere. The load is then applied to rectangular strips, $4 \times \frac{1}{2}$ in., of the films. The representative values found are as follow: a high-grade commercial glue, 12,200—12,400; high-grade commercial gelatin, 12,800; purified gelatin, 9500; refined isinglass, 9500; and glue size, 7200 lb./in.^{2} The tensile strength of such films depends on the humidity of the atmosphere. The effect of an addition of a powdered solid on the resulting strengths of the films of glue depends on the quantity added, *e.g.*, 10—15% by weight of lead sulphate has no effect, but an addition of 66·7% weakens the tensile strength by 31%. A similar loading of the glue by zinc oxide, aloxite, and fine water-ground flint (fired) caused an even greater weakening; of the soluble substances added to the glue, only potassium dichromate increased the strength, whilst formalin had no effect; potash alum, sodium formate, salicylate, or benzoate, and glucose reduced the strength. Films prepared from 25% solutions of glue in water or 0·1*N*-hydrochloric acid were equally tenacious, whereas those with 0·1*N*-sodium hydroxide were decidedly weaker. The hydrolytic degradation of such aqueous, acid, and alkaline solutions of glue continuously heated brings about a relatively slight loss in strength after 28 days at 55—57·5°, but at higher temperatures (up to 100°) the effect is much greater, and films, even when of high tensile strength, become very brittle. The gelatin, glue, and isinglass films, when loaded nearly to the point of fracture, developed permanent, opaque markings suggestive of slip planes or crystallisation of the adhesive. *X*-Ray examination of one of these films gave no indication of crystalline structure, however. The paper strip method of Millar (J.S.C.I., 1899, 16) is shown to be unsatisfactory even as a method for comparing the tensile strengths of different adhesives. The experimental error involved in the present method is generally much less than 10%, and it is claimed that the method will supplement and, to some extent, supersede the more indirect methods now in use. L. S. THEOBALD.

PATENT.

Tanning preparations [from sulphite-cellulose waste lye]. T. BLACKADDER, Assr. to ROBESON PROCESS CO. (U.S.P. 1,571,873, 2.2.26. Appl., 10.6.24).—Sulphite-cellulose waste liquor is treated with slightly more lime than is required to neutralise it, the precipitate is separated, the liquor concentrated, and the lime in solution is precipitated with sulphuric acid. 1·25—2·0% of lime is used for a waste liquor requiring 0·5% for neutralisation. D. WOODROFFE.

XVI.—AGRICULTURE.

Mechanical analysis of soils. Report on the present position and recommendations for a new official method. SUB-COMMITTEE OF THE AGRICULTURAL EDUCATION ASSOC. (J. Agric. Sci., 1926, **16**, 123—144; cf. Robinson; B., 1922, 990A).—The method hitherto in use (J. Agric. Sci., 1906, **1**, 470) does not secure a complete preliminary dispersion of the sample. The method recommended as official includes preliminary treatment with hydrogen peroxide, to remove humified organic matter, and with $0\cdot2N$-hydrochloric acid to remove calcium carbonate and adsorbed bases. Dispersion is effected by shaking in ammoniacal water. The actual analysis is effected by the pipette method, depending on the depth-concentration relationship. Full working details are given.
G. W. ROBINSON.

Evaporation of water from soil. B. A. KEEN, E. M. CROWTHER, and J. R. H. COUTTS (J. Agric. Sci., 1926, **16**, 105—122; cf. Keen, B., 1915, 146).—Further investigations on the evaporation of water from soil are reported. The drying of moist soil in shallow pans is irregular, and more readily reproducible results are obtained by exposing the soil in thin layers below glass plates. During the early stages, drying is largely confined to the peripheral portions. Evaporation of water from soil is controlled by two groups of factors depending respectively on soil-water relationships and on environmental conditions. The latter include such factors as diffusion of water vapour, bulk air movements, and temperature gradients. The precise interpretation of evaporation curves cannot be attained unless a carefully controlled and reproducible technique is used. G. W. ROBINSON.

Relations between certain soil moisture constants and the determination of the vesicular coefficients of soils. C. H. WRIGHT (J. Agric. Sci., 1926, **16**, 18—23; cf. Hardy, B., 1923, 846A).—The author discusses Wilsdon's modification of the Briggs-Shantz formula connecting the maximum water-retaining capacity with the hygroscopic coefficient (cf. B., 1925, 19). Evidence is produced to show that the content of free water in a soil at the maximum water-retaining capacity is 21%. The values obtained by the author for the vesicular coefficient are in good agreement with those obtained by Hardy from the moisture content at point of maximum plasticity. The vesicular coefficient is greater for soils than for subsoils. The percentage of vesicular water is equal to the cubical shrinkage coefficient. G. W. ROBINSON.

Anomalous flocculation in colloidal clays and soils. F. HARDY (J. Physical Chem., 1926, **30**, 254—265).—Theoretical. Flocculation phenomena in lateritic clays and soils are discussed in the light of Comber's theory (Trans. Faraday Soc., 1922, **17**, 349; cf. B., 1922, 69A), which attributes the anomalous flocculation of clay by lime to the formation of insoluble colloidal complexes. Hydrous alumina and hydrous ferric oxide are considered to be colloidal ampholytes, possessing isoelectric points not far removed from neutrality, and the mechanism of their anomalous flocculation by lime and by anions such as the silicate and phosphate ions is explained on this view. The theory is extended to hydrous silica, hydrous aluminosilicates, and ferrosilicates. These may also be regarded as ampholytes. Mutual precipitation is discussed in relation to flocculation in certain soils. L. S. THEOBALD.

Action of some antiseptics on soil amœbæ in partially sterilised soils. L. SEWERTZOV (Zentr. Bakt. Parasitenk., Abt. II., 1925, **65**, 278—291; Chem. Zentr., 1926, I, 421).—The effect on soil amœbæ of a number of sterilising agents was determined. The fatal dose of easily soluble antiseptics was much smaller for organisms in culture solutions than for the same organisms in the soil. The fatal dose for amœbæ in soil is so great as to place these organisms beyond consideration in partial sterilisation phenomena. Spores of soil bacteria are more resistant to antiseptics than are amœbæ cysts but non-sporing bacteria have a lower resistance. Treatment of sterilised soils with 0·5—1·5% of calcium sulphide has little effect on the number of amœbæ, but causes a decided reduction in the number of bacteria. Treatment of sterilised soil with 5% of calcium sulphide leads to an initial decrease in the number of amœbæ, followed by an increase in the numbers of both amœbæ and bacteria. Treatment of a normal soil with 1·5% of calcium sulphide has a negligible effect on the number of amœbæ. Partial sterilisation effects cannot be attributed to reduced numbers of amœbæ. A. G. POLLARD.

Age of seedlings as a factor in the resistance of maize to sodium chloride. G. J. HARRISON and C. J. KING (J. Agric. Res., 1925, **31**, 633—639).—The resistance of germinated maize seedlings to solutions of sodium chloride depends on the degree of development, the young plants with radicles 25 mm. long and no plumules being the least resistant, the old plants with two developed plumules coming next, whilst those of intermediate age show the greatest resistance. Artificial excision of the radicles increases the resistance, which is also influenced by the weather conditions.
W. O. KERMACK.

Determination of perchlorate in Chili saltpetre based on precipitation with methylene-blue. K. A. HOFMANN, F. HARTMANN, and U. HOFMANN (Ber., 1925, **58**, 2748—2754).—In consequence of its toxic action towards plant life the limit of perchlorate, calculated as potassium perchlorate, in Chili saltpetre is fixed at 0·5%. Percentages of this order of magnitude are rapidly determined by dissolving 20 g. of the sample of nitrate to 100 c.c. in water and adding 10 c.c. of this solution to 5 c.c. of 0·1% aqueous methylene-blue B extra; the time requisite for the first appearance of a precipitate is noted. This with 0·6% $KClO_4$ in the saltpetre is 1—2 sec., with 0·5%, 10 sec., with 0·4%, 40 sec., and with 0·3% 90 sec. at atmospheric temperature.

As control, the solutions are filtered after 10 min. and the filtrates are absorbed in equal strips of white filter-paper, which are then drained and dried at 100°. The depth of colour of these papers is compared with that of standard papers prepared by use of saltpetre of known perchlorate content from 0·3 to 0·6%. With 0·6% $KClO_4$ the paper is almost white, with 0·5% just visibly bluish, with 0·4% distinctly and with 0·3% strongly pale blue in colour. If the perchlorate content is above 0·5% the method may be applied to a suitably diluted solution. In the region of 0·5% $KClO_4$ the results are accurate to +0·05%. If the perchlorate content lies near the limit it is advisable to compare the colours of the filtrates described above with those of methylene-blue solutions of known content and to refer to curves obtained by use of artificial mixtures of potassium perchlorate and sodium nitrate. Closer examination of the precipitate shows that it contains the nitrate in addition to the perchlorate of methylene-blue. With solutions containing up to 10% of pure sodium nitrate the precipitation of methylene-blue as nitrate from the chloride of the dye is proportional to the solubility product of the nitrate, whereas at higher concentrations salting out is also evident. For this reason methylene-blue chloride is unsuitable for the determination of very small amounts of perchlorate in Chili saltpetre, but it may be replaced by a solution of the perchlorate of the dye prepared by mixing 320 c.c. of 0·1% methylene-blue B extra solution with 250 c.c. of 0·1% potassium perchlorate solution, filtering, and washing the precipitate with 3×10 c.c. of water. 5 c.c. of a solution of 40 g. of sample made up to 100 c.c. with water are mixed with 5 c.c. of this solution and the time is taken until the first precipitate appears; with 0·2%, 0·15%, and 0·1% of potassium perchlorate in saltpetre the times are 9, 15, and 90 sec.; with 60% saltpetre solutions the times for 0·1%, 0·08%, and 0·06% $KClO_4$ are 20, 45, and 120 sec. respectively. Controls are effected exactly as described for methylene-blue B extra solutions. The presence of chlorate (up to 1%), iodate, periodate, or chloride in the concentrations observed with technical, crystallised Chili saltpetre has no influence on the results and the ordinary variations in atmospheric temperature need not be taken into account. H. WREN.

Petroleum insecticides. G. P. GRAY and E. R. DE ONG (Ind. Eng. Chem., 1926, **18**, 175—180). —Toxic action of petroleum oils on plant life takes the form of defoliation, stunted growth, and deterioration in the quality of fruit, and in general has been found to follow the use of unrefined oils as a spray. Representative samples of petroleum oils were examined with a view to correlate their physical and chemical properties with their efficiency as insecticides. Further samples were also obtained by bulking the first samples, redistilling, and collecting fractions at 25° intervals. Tests on foliage and fruit were carried out by spraying orange, citrus, and black walnut trees with these oils in the form of soap emulsions of various strengths. In general the heavier oils, *i.e.*, the higher-boiling fractions, are the most toxic to plant life, but this property is more clearly indicated by the amount of unsaturated hydrocarbons present. The oil most soluble in concentrated sulphuric acid is the most toxic in its action, and the worst oil in this respect was a sample recovered from the acid tar formed in the treatment of kerosene with sulphuric acid. A test is described for the determination of the solubility of the oil in 37*N*-sulphuric acid, together with a simple test for showing comparative capillarity. A table showing the characteristics and action of all the oils examined is given. S. BOWMAN.

Solid calcium cyanide. METZGER.—See VII.

PATENTS.

Calcium nitrate fertiliser. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 246,377, 22.6.25).—Concentrated calcium nitrate solutions are mixed with urea in the proportion of 1 mol. of the former to 1—4 mols. of the latter, and the heated solution is forced through a spray nozzle, *e.g.*, by compressed air, to obtain a granular product which is readily distributed.

A. G. POLLARD.

Insecticides, sheep dips and the like. McDOUGALL and YALDING, LTD., and P. J. FRYER (E.P. 246,252, 12.11.24. Addn. to 229,773; cf. B., 1925, 334).—Sulphonated castor oil specified in the earlier patent may be replaced by any other sulphonated fatty oil, sulphonated sperm oil, or sulphonated wax. The active principles of derris are extracted with benzene or other solvent, and after distilling off the solvent the residue is dissolved in sulphonated oil or wax. Calcium salts of the sulphonated oils may be used in some cases. Lime-sulphur may be mixed with the insecticide without precipitation if a proportion of a colloid, *e.g.*, casein, is added. A. G. POLLARD

Composition of matter. [Insecticide.] H. P. BASSETT and M. R. ISAACS, Assrs. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,568,044, 5.1.26. Appl., 1.11.22).—Calcium diarsenate (70 pts.), calcium oxide (30 pts.), and casein (3 pts.) are mixed in a moist condition to form a product which when sprayed on plants or the like, either dry or with water, adheres for long periods owing to the adhesive action of the calcium caseinate formed. The caseinate also coats the arsenate particles, so that the insecticidal and vermicidal properties of the mixture are preserved. T. S. WHEELER.

Manure from sewage (E.P. 224,226).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Hydroxyl-ion concentration and volumetric alkalinity in beet juice clarification. A. H. W. ATEN and P. J. H. VAN GINNEKEN (Tijdschrift; Int. Sugar J., 1926, **28**, 54; cf. B., 1925, 684, 1014). —Using a form of hydrogen electrode potentiometer, the p_H values were determined for juices after the

first and second carbonatations, and for the carbonated juice after sulphitation, comparison being made with an artificial juice consisting of a solution containing 12·5% of sucrose and 1% of potassium chloride to which lime had been added. At the same volumetric alkalinity the p_H values of ordinary limed juice were considerably lower than those of the artificial juice containing lime only as base. At the end of the first carbonatation, the p_H value was not very much lower than that of the limed artificial juice; after the second carbonatation the difference was greater; and after sulphitation it was greater still. J. P. OGILVIE.

Behaviour of raffinose in the process of crystallisation of sugar. R. MEHRLE (Deuts. Zuckerind., 1925, 50, 1325—1328, 1357—1361).—Sugar crystallising out from massecuites containing raffinose may contain 40—50% of this trisaccharide, 5—10% of ash, and 45—50% of organic non-sugars reckoned on the non-sucrose substances present. Generally about one-third of the raffinose originally present crystallises out with the sugar, this amount depending on whether raw or white sugar is being made. Raffinose has a considerable influence on the yield of crystals obtained in refining raw sugars.
J. P. OGILVIE.

Decolorising carbons [for sugar refining]. A. A. BLOWSKI and J. H. BON (Ind. Eng. Chem., 1926, 18, 32—42).—A number of commercial decolorising carbons were found all to show moderate or high decolorising power and ash-adsorptive properties, but certain of them proved poor filtering media, making them unsatisfactory for practical use in the sugar refinery. Laboratory-scale tests indicated that by the use of decolorising carbon it would be possible to produce a granulated sugar which compares favourably with that made by the bone-char process. The nett cost of the two processes would, however, be about the same, and in view of the difficult practical problems which would require solution, no advantage would be gained by substituting the former for the latter and older mode of refining. A combined carbon-and-char process is considered to be an undesirable substitute for the simple bone-char process.
J. P. OGILVIE.

Determination of mixtures of sucrose and invert sugar or lactose. II. H. JESSEN-HANSEN (Compt. rend. Trav. Lab. Carlsberg, 1925, 16, [4], 1—7).—Work on this subject previously published (B., 1923, 1237 A) has been elaborated, the procedure being modified so that the action of hydrochloric acid on the lactose is reduced to a minimum, and so that the method may be applicable when sucrose is present in notable excess. Moreover, the tables have been extended. J. P. OGILVIE.

Detecting coal tar dyes in caramel. L. VON NOËL (Pharm. Zentr., 1926, 67, 33—35).—For the detection of aniline dyestuffs in commercial caramels it is essential that very dilute solutions be used and that double dyeing be adopted, *i.e.*, that a second thread is dyed by means of the colour on the first coloured thread. As a fixing agent tartaric acid is to be preferred to potassium sulphate.
J. P. OGILVIE.

PATENTS.

Refining of saccharine liquids. P. SINGH and S. K. S. MAJITHIA (E.P. 244,924, 6.1.25).—A process for clarifying or decolorising saccharine liquids consists in removing suspended impurities, rendering the liquid alkaline, adding aluminium salt and alkali to form aluminium hydroxide, heating to about 100°, and separating the precipitated matter. A bleaching and clarifying agent which under heat will decompose into a metal hydroxide and nascent sulphur dioxide (*e.g.*, aluminium hydroxide suspended in sulphur dioxide solution) is added to the separated liquid after it has been rendered acid, the mixture being boiled, and the precipitated matter finally separated. In the first filtration kieselguhr may be added to form easily washed cakes.
J. P. OGILVIE.

Manufacture of sugar. A. P. LEONARD (U.S.P. 1,568,512, 5.1.26. Appl., 26.7.21).—Bagasse is passed successively through a series of mills, or crushing units, and the juice is conveyed from the foremost of them to separators, where clear juice is extracted. Sugar is separated from the clear juice from one separator, and water is introduced into the residuum in another separator. The clear juice from the separators is then returned to an intermediate mill of the series, juice from these intermediate mills being passed to the foremost mill. Juice from the last mill of the series is sent to a mill in advance of that receiving the liquid from the separators, whilst that from the advance mills goes to the foremost mill. J. P. OGILVIE.

Recovery of sugar from molasses. G. R. BLUM (F.P. 594,526, 10.5.24).—Molasses is mixed at ordinary temperature with 60—90% of a fatty acid, or an alcohol, or a hydrocarbon, the mixture kept for several hours, and the sugar then separated from the liquor above it, from which potassium salts, nitrogenous substances, and the added substances can later be recovered.
J. P. OGILVIE.

Making grape sugar. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,571,212, 2.2.26. Appl., 28.5.23; cf. U.S.P. 1,508,569; B., 1924, 1024).—Starch is converted into dextrose, and the aqueous solution is supersaturated by heating and cooled to, and maintained at, a temperature favourable to production of crystals of anhydrous dextrose. These are separated, and the mother-liquor is cooled further to induce production of hydrated crystals, which are then separated from the mother-liquor. D. G. HEWER.

Extraction of total sugar from apples and pears. DISTILLERIES DES DEUX-SÈVRES (SOC. ANON.) (F.P. 596,201, 12.7.24).—The fruits are subjected to a single or repeated pressing and the residue is treated in a diffusion battery. The juice

obtained is worked up for sugar by the known methods. E. H. Sharples.

Treatment of condensate obtained by distillation of carbohydrate solutions containing hydrochloric acid. T. Goldschmidt A.-G., Assees. of E. Hägglund and E. Färber (G.P. 421,829, 19.7.22).—The condensate containing hydrochloric and acetic acids is treated with a dehydrating agent, *e.g.*, sulphuric acid, phosphoric acid, or calcium chloride, at a temperature below the boiling point and the issuing vapours are collected. The residue after expelling hydrochloric acid is heated to boiling to obtain the acetic acid. E. H. Sharples.

Manufacturing yeast (E.P. 246,002).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Loss of alcohol in spirits. H. Lührig (Pharm. Zentr., 1926, 67, 49—55).—There may be an apparent loss of alcohol due to an increase of the specific gravity of the spirit by extraction and solution of matter from the (wooden) container. When freshly scalded containers are used a diminution of from 0·1 to 0·15% in the alcohol content may be caused by extraction of water from the pores of the wood. The contents of part-filled casks, subject to fluctuation of temperature, may not be homogeneous, the upper portions being the richer in alcohol. Differences of 4·4% and 0·4% of alcohol (by volume) respectively were found in two brandies which had been in cask for 7 months and for 2¾ years. The same action may take place, on a smaller scale, in the retailer's bottle. No loss of alcohol was observed in brandy kept in bottles (for four months) the stoppers of which were removed for a minute four or five times a week. Brandy kept in an open bottle for a month lost 0·5—0·7% of alcohol. A margin of 1% of alcohol should be allowed for both by the distiller and by law, to cover possible losses of alcohol during storage and sale. The literature of the subject is reviewed. G. T. Peard.

See also A., Mar., 274, **Specific action of α-glucosidase from yeast** (Helferich, Klein, and Schäfer). 321, **Isolation of enzymes** (Willstätter); **Amylase action of emulsin preparations** (Josephson); **Milk-diastase** (Chrzaszcz and Goralowna); **Decrease of invertase in yeast** (Willstätter and Lowry, jun.); **Invertase** (Willstätter, Schneider, and Wenzel). 322, **Enzyme action and ion antagonism** (Höber and Schürmeyer); **Maltase of barley malt** (Leibowitz); **Hitherto undescribed oxydase in certain fungi** (Wolff); **Cell-free fermentation of hexose-diphosphoric acid, dextrose, lævulose, sucrose, and invert-sugar** (Neuberg and Kobel). 323, **Nomenclature of fermentation enzymes and oxydases** (Neuberg and Oppenheimer); **Dehydrogenases of yeast** (Von Euler and Nilsson); **Hydrolysis of polysaccharides by yeast** (Iwatsuru).

Patents.

Process of manufacturing yeast. H. W. Dahlberg (E.P 246,002, 20.5.25).—The waste water produced in the process of extracting sugar from beet molasses and syrups, which contains amino-nitrogen, is added to the yeast mash, so as to supply from 0·4 to 1·0 pt. of amino-nitrogen to 100 pts. of sugar in the mash. The waste water is alkaline, and is added at intervals in quantities depending on the p_H of the mash and is thus used both to supply nitrogen and to control the acidity during the growth of the yeast. C. T. Gimingham.

Preparation and application of an adsorption agent for the purification of alcohols and oils. Rieri-Werk E. Richter (G.P. 422,313, 14.6.21).—The product obtained by calcining a mixture of silica, aluminium hydroxide, and lime-wood charcoal is agitated with the liquid to be purified and then separated. Removal of empyreumatic substances, fusel oil, and substances producing turbidity is thus quickly effected without injury to the product. E. H. Sharples.

XIX.—FOODS.

Determination of the ash-content of flour. A. Goske (Z. Unters. Nahr. Genussm., 1925, 50, 428—429; of B., 1925, 778).—A weighed quantity of sample is made into a paste with water and about twice its weight of powdered pumice (previously heated and weighed) in a platinum dish. The paste is dried, heated gently, and after the char has been crushed, is placed in a muffle for 15 min. at a moderate temperature, cooled, and again weighed. J. Grant.

Relation between the bacterial count of whole milk and that of the cream and skim milk separated from it. C. S. Leete (J. Agric. Res., 1925, 31, 695—699).—Cream obtained by a clean centrifugal separator does not give a greatly higher bacterial count than the whole milk from which it is derived, whereas cream separated by gravity gives a much higher bacterial count than the whole milk. W. O. Kermack.

Alkaline milk and its detection by the bromocresol-purple test. I. F. Procter and A. T. R. Mattick (J. Agric. Sci., 1926, 16, 145—148).—Directions are given for the testing of milk for alkalinity by the use of bromocresol-purple test papers. Results are given for samples of milk from a number of cows at different dates. In 92·8% of the cases examined, the reaction was between p_H 6·5 and 6·75, whilst in 3·3% of the cases the reaction was between p_H 6·9 and 7·13. G. W. Robinson.

Determination of palm kernel oil and butter fat in margarine. G. D. Elsdon and P. Smith (Analyst, 1926, 51, 72—76).—In continuation of earlier work (B., 1925, 224), Reichert–Polenske–Kirschner figures have been determined for various mixtures of palm kernel oil, butter fat, and other vegetable oils, and tables of corrections are given for

deducing the true figure for each value from the observed. The Reichert values are 0·3 unit too high when no butter is present and correct when 10% is present. The Kirschner values are corrected by deducting 0·1 when the butter content is small. The Polenske corrections are irregular.

A. R. POWELL.

Apple-juice and commercial pectin products. H. ECKART (Z. Unters. Nahr. Genussm., 1925, 50, 405—415).—A number of analyses of raw and boiled apple-juices and of commercial pectin products have been carried out, the density, refractive index, acidity, total solids, viscosity, and pectin and starch contents being determined. Straight lines are obtained when these values are plotted against the concentration of the juice at different stages of evaporation. An attempt is made to assess the commercial value of a product by means of its "pectin-value," *i.e.*, pectin content × 100/percentage extract, the pectin being determined by the calcium pectate method. The influence of concentration on the pectin-value and jelly-forming capacity is also discussed, and some additional criteria are suggested for the assessing of commercial products.

J. GRANT.

Distinguishing reconstituted dried preserved peas from preserved green peas. C. F. MUTTELET (Compt. rend., 1926, 182, 238—240).—Preserved green peas have the same chemical characteristics as fresh peas, viz., ratio of starch to soluble sugars 5·1:1; starch to cellulose, 4·1:1; nitrogen content of insoluble constituents to nitrogen content of soluble constituents, 3·8:1. Reconstituted dried peas have a different composition; the ratio of starch to soluble sugars is 11·1 : 1; starch to cellulose, 7·6 : 1; insoluble to soluble nitrogen, 9·6:1. The reconstituted dried peas, therefore, have a higher proportion of insoluble constituents, due, it is suggested, to the cooking process they undergo during reconstitution.

L. F. HEWITT.

Determination of husk in cocoa. C. H. KOPERBERG (Chem. Weekblad, 1926, 23, 64—66; cf. Silberberg, B., 1923, 26 A).—2 mg. of the cocoa are defatted by treatment successively with nitric acid and potassium hydroxide, the liquids being separated in each case by centrifuging, and the residue is examined microscopically. The husk cells are counted, and the results compared with those obtained from pure dehusked and defatted cocoas to which known proportions of husk have been added. Material which has been passed through the bolting mill loses a proportion of its husk. S. I. LEVY.

Study of the process of making stack silage. H. E. WOODMAN and F. HANLEY (J. Agric. Sci., 1926, 16, 24—50; cf. Amos and Woodman, B., 1925, 1008).—An account, with temperature readings and chemical data, of experiments on stack ensilage. During the ensilage two distinct temperature maxima occur. The significance of the second maximum is not certain, but the evidence points to anaerobic cellulose bacteria as being responsible. The losses of dry matter in the "sweet" silage layer compare favourably with those obtained in tower ensilage and are appreciably smaller than those involved in the production of "sour" silage in the stack. The initial high temperatures largely inhibit the formation of amino-acids from proteins owing to the destructive effect on the plant enzymes. Ether-soluble material decreases during ensilage. The decrease in digestibility of the protein is largely due to changes similar to humification; a similar explanation may serve for the decreased protein digestibility of heated brown hay. The destruction of carbohydrates cannot be correlated with the temperatures attained during fermentation. G. W. ROBINSON.

Sunflower as a silage crop—composition and yield at different stages of maturity. W. L. GAINES and W. B. NEVENS (Univ. Illinois Agric. Exp. Sta. Bull., 1925, [268], 407—455; Chem. Abstr., 1926, 20, 78).—Sunflowers produce greater yields of dry matter per acre than maize; they contain more ash, crude fibre, and crude fat, but less nitrogen-free extract. A sunflower crop removes about 900 lb. of ash from 1 acre of soil. The relative proportions of the constituents of the ash are approximately constant, but not that of the ash and the organic matter. A. A. ELDRIDGE.

Dry matter of swedes. I. F. W. SANSOME (J. Agric. Sci., 1926, 16, 51—58).—Mainly an investigation of methods of sampling swedes for analysis.. There is a well marked inverse correlation between dry matter content and size of root.

G. W. ROBINSON.

Modern views on vitamins. J. C. DRUMMOND (J. Soc. Arts, 1926, 74, 369—388).

Vitamin potency of cod-liver oils. HOLMES and PIGOTT.—See XII.

PATENTS.

Drying and congelation of egg albumins. E. E. LIBERSA (E.P. 229,669, 18.2.25. Conv., 22.2.24). —Certain alkaline-earth hydroxides or basic salts, *e.g.*, calcium hydroxide or calcium saccharate, are added in the proportion of about 5 pts. per 1000 to liquid white of eggs before drying or congelation. The albumin may then be dried directly, without preliminary fermentation and addition of ammonia; and all the properties of fresh egg albumin are retained. C. T. GIMINGHAM.

Puffed [food] products. ANDERSON PUFFED RICE CO., Assees. of A. P. ANDERSON (E.P. 232,543, 21.7.24. Conv., 21.4.24).—The material, *e.g.*, cereal food, containing moisture and/or matter which expands greatly on reducing the pressure, *e.g.*, a gas, is reduced to such a form that it flows under pressure. From the pressure vessel, after impregnating or not with carbon dioxide, it is passed under a pressure of 500 lb. through a heater, which is a steam jacket containing a number of tubes leading to an orifice or orifices, the total cross-sectional area of the orifices being less than that of the tubes. The material is then passed through a drying chamber in which the

heat preferably decreases in intensity in the direction of travel. A puffed product made thus consists of a strip of cellular formation, the walls of the cells containing disrupted starch granules. The outer cells are smaller and have thicker walls than the inner, and the centre of the strip is hollow.

D. G. HEWER.

Spray drying apparatus for manufacturing milk or like powder. MILKAL, LTD., and J. M. SIERRA (E.P. 246,239, 3.11.24).—Milk or the like is concentrated and then fed under pressure to the atomising nozzles, by a pump provided with a by-pass containing a loaded relief valve capable of adjustment so that return of pressure liquid to the suction side of the pump may take place when a definite pressure is attained. With this arrangement the milk may be pre-concentrated to a higher degree than is usual; or the use of additional heat to reduce the viscosity of the concentrated milk may be avoided.

D. G. HEWER.

Filter, especially applicable to milk and other potable liquids. R. SELIGMAN (U.S.P. 1,568,001, 29.12.25. Appl., 13.5.25).—The filter comprises a fixed median plate fitted on both sides with cover plates so constructed that filter chambers are formed between them and the median plate, the chambers being divided by means of filtering media into spaces for filtered and unfiltered liquid, respectively. Supply of liquid through one or other of the filter chambers is controlled by means of a multi-way valve on the median frame, the valve being connected with the filter chambers through the median plate and through projections on the cover plates.

L. A. COLES.

Vitamin [malt] preparation. SOC. OF CHEM. IND. IN BASLE, Assees. of O. STINER, A. HAUSWIRTH and A. GAMS (U.S.P. 1,568,196, 5.1.26. Appl., 18.1.19).—Amylaceous material rich in vitamins is mashed and saccharified with ground, fresh, green malt at temperatures below 60°, and sufficient of a non-toxic polybasic organic acid is added to make the mash feebly acid to Congo-red. The filtered mash is evaporated to the desired consistency. The preparation is in a durable form and contains both heat-stable and heat-labile vitamins.

G. T. PEARD.

Production of caffeine-free coffee. F. KÜNDIG, JUN., & Co. (F.P. 597,392, 28.4.25. Conv., 6.5.24).—The raw, whole coffee-beans are treated in an autoclave under 22 atm. pressure, with hydrocarbons, ketones, or alcohols, and alkalis or organic acids.

J. GRANT.

Production of caffeine-free coffee. SCHWEIZER UND Co. (Swiss P. 100,167, 4.12.22).—The raw coffee-beans are treated with 1% ammonia solution *in vacuo*, and extracted with acetone. J. GRANT.

Baking powders. C. H. BOEHRINGER SOHN, CHEM. FABR., Assees. of F. RAHN (G.P. 418,341, 20.7.22).—The rising-power of baking powders prepared from lactic acid and calcium lactate is increased by the addition of comparatively small quantities of mono- or di-basic phosphates so that the total acidity of the powder is not reduced by more than one-half. Such baking powders yield results similar to those obtained by the use of yeast. A. J. HALL.

Electrical apparatus for preserving sap-containing fodder. SIEMENS-SCHUCKERTWERKE G.m.b.H., Assees. of O. KAUFMANN and E. CRAMER (G.P. 418,481, 10.11.21).—The consumption of electrical energy in the treatment of green fodder is considerably reduced by using a number of electrodes to which the current is supplied separately so that on introducing fresh fodder, the current can be cut off from that part of the container containing the fodder which has already been subjected to treatment.

A. J. HALL.

Manufacture of preparations which evolve carbon dioxide when heated or dissolved in water. J. D. RIEDEL A.-G. (G.P. 418,485, 15.1.21).—Effervescent preparations which yield refreshing beverages having a pleasing flavour are prepared from adipic acid or its salts and ammonium or alkali carbonates; they are not hygroscopic and are thus superior to the usual preparations which contain tartaric acid or its salts. A. J. HALL.

Manufacture of a foodstuff [from yeast]. C. ENGEL (G.P. 421,707, 18.9.23. Addn. to 413,414).—By raising the temperature to 180—220°, yeast treated as described in the previous patent (B., 1925, 735) is converted into a paste suitable for spreading on bread. J. GRANT.

Production of high-grade flour from maize. J. HÄUSLER (G.P. 421,870, 6.10.18).—Dry coarse-ground maize is freed from embryos by washing in water, and steeped in an acid solution at 70—90°. After neutralisation and drying, the meal is separated by any convenient means and finely ground.

J. GRANT.

Production of high-grade flour from maize and other cereals. J. HÄUSLER (G.P. 421,871, 21.12.18).—The material is converted into a paste by means of an acid solution, preferably a solution of hydrochloric acid, and is then atomised and carried by a current of dry air through a chamber in which the flour-, gluten-, and bran-ingredients separate and are deposited in different parts of the chamber according to their densities. J. GRANT.

Conversion of hard cheese into durable soft cheese. M. CLAASS (Austr. P. 100,222, 5.3.24).—Hard cheese is heated for a short time at a temperature not exceeding 100° with neutral alkali phosphates. A. J. HALL.

Manufacture of margarine and edible fats. E. V. SCHOU (U.S.P. 1,570,529, 19.1.26. Appl., 14.10.21).—See E.P. 187,299; B., 1922, 994 A.

Manufacture of albumins and fatty matters. M. KAHN, Assr. to SOC. FRANÇ. DES PROD. ALIMENTAIRES AZOTÉS (U.S.P. 1,572,933, 16.2.26. Appl., 14.4.25).—See E.P. 236,158; B., 1925, 939.

Fats (G.P. 422,687).—See XII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of atropine. P. Bourcet (Bull, Sci. Pharmacol., 1925, **32**, 585—588; Chem. Zentr.. 1926, I., 1465).—A mixture of 100 g. of the powdered drug, ground to pass through an 80-mesh sieve, with 20 g. of finely powdered calcium oxide, is extracted with ether in a Soxhlet apparatus. The ether extract is shaken with 2% hydrochloric acid, and the aqueous layer is removed, filtered, rendered alkaline with sodium hydroxide, and extracted with chloroform. After filtering the chloroform solution through a layer of anhydrous sodium carbonate, the solvent is evaporated off, and the residue is dried at 100° and weighed, giving the total weight of non-volatile alkaloids. After heating to 120° for 5 hrs. in order to convert hyoscyamine into its isomeride, the alkaloid mixture is dissolved in absolute alcohol, and the atropine is precipitated as oxalate by the addition of an ethereal oxalic acid solution, the precipitate being removed, washed with ether, dried at 100°, and weighed. L. A. Coles.

Use of hexylresorcinol and its homologues as internal antiseptics. V. Leonard (Science, 1925, **62**, 408—412; Chem. Zentr., 1926, I., 1231).—The lower alkylresorcinols possess strong bactericidal properties, and are relatively non-poisonous, the antiseptic properties increasing and the poisonous action decreasing in proportion to the sum of the atomic weights of the alkyl radicals. The maximum bactericidal action is reached in hexylresorcinol, which has a phenol coefficient of 46—56·3. This compound, and other higher members of the series, have been prepared as described in E.P. 219,922, 223,190, and 224,913 (cf. B., 1925, 378, 568). No poisonous symptoms developed when hexylresorcinol was given to dogs in repeated per-oral doses of 0·5 g. for 21 days, or in single doses of 1 g. per kg. The compound causes no irritation of the urinary ducts, develops its bactericidal action when strongly diluted with urine of suitable reaction, and partially separates in the kidneys in the form of a non-reactive conjugation product, enough remaining uncombined, however, to exert bactericidal action in the urine. It is suitable for use in treating cases of infection of the urinary ducts, such as pylitis and cystitis. L. A. Coles.

Volatility of salicylic acid. A. F. Lerrigo (Analyst, 1926, **51**, 79).—The salicylic acid content of an ethereal solution obtained by extracting medicinal substances with methylated ether may be determined directly by allowing the ether to evaporate spontaneously and drying the residue at room temperature for 48 hrs., or preferably for 2 hrs. in an oven at 35°. Salicylic acid does not volatilise appreciably below 40°, but between 50° and 60° volatilisation is comparatively rapid. A. R. Powell.

Preparation of aspartic acid from asparagine. F. Pachlopnik (Z. Zuckerind. Czechoslov., 1925, **50**, 139—141).—Asparagine is hydrolysed by dilute nitric acid, excess of acid neutralised by ammonia, the liquid evaporated to dryness, and ammonium nitrate extracted from the residue by hot alcohol. J. P. Ogilvie.

Pine oil and the products of its autoxidation. P. Epple and S. Ruhemann (Ber., 1926, **59**, 294—301).—The methods which have led to the isolation of azulene from the neutral oil of lignite tar (Herzenberg and Ruhemann, B., 1926, 42) have been applied to pine oil. The pine oil, which had been preserved for about ten years, was subjected to a preliminary treatment with dilute sulphuric acid and alkali and fractionally distilled under diminished pressure. The separate fractions were treated with hydroferrocyanic acid and the autoxidation products separated from the precipitates by extracting the latter with ether; the residual precipitate was treated with sodium carbonate at atmospheric temperature and the separated oil was isolated, after which the aqueous solution was boiled and the hydrolytically separated alcohols were extracted; the alkaline solution which remained contained the organic acids which, however, could not be separated from admixed hydroferrocyanic acid. The presence of camphor, borneol, cineole, terpin hydrate, and *p*-tolyldimethylcarbinol was thus established. These substances are not present in the oil previous to autoxidation. Terpin hydrate does not exist in the oil, its production being due to the action of hydroferrocyanic acid on terpineol. H. Wren.

Anise oil and star-anise oil. W. Zimmermann (Apoth.-Ztg., 1925, **40**, 1344—1345; Chem. Zentr., 1926, I, 1310).—The hydrochloric acid test with pure anise oil, star-anise oil, and mixtures of the two does not give trustworthy results and the following is suggested. Five drops of a vanillin solution (0·4 g. of vanillin in 5 g. of dilute alcohol) are mixed with 2—3 drops of the oil, and fuming hydrochloric acid is added to make 1 c.c. The colour is first observed in the cold, then in a water bath at 50°, which is slowly heated to boiling. Freshly distilled anise oil becomes pale red on warming and finally brownish-red, which remains on cooling. Star-anise oil on warming gradually becomes pale green, then grass-green, and on boiling brownish-green; on cooling, olive green. With a mixture of anise oil and 10% of star-anise oil a dirty green colour is obtained, and with 30% of star-anise oil the colour produced by the anise oil is entirely masked. E. H. Sharples.

Manufacture of alcohols from hydrocarbons with particular reference to petroleum as a raw material. B. T. Brooks (Chem. Reviews, 1926, **2**, 369—394).

Poisoning by chloronitrobenzene and acetanilide. Renshaw and Ashcroft.—See IV.

See also A., Mar., 235, **Partial formaldehyde vapour pressure of aqueous formaldehyde** (Ledbury and Blair). 236, **Separation of mixed liquids by action of salts** (Sandonnini and Gerosa). 238, **Quinine hydrochloride and**

antipyrine (Oliveri-Mandalà and Carli); **Acetanilide–antipyrine and acetanilide–pyramidone** (Oliveri-Mandalá and Forni). 250, **Catalytic activity of titania in decomposition of alcohols** (Rudisill and Engelder). 286, **Organic peroxides** (Gelissen and Hermans); **Benzoyl hydroperoxide** (Levy and Lagrave). 288, **New benzyl compound [betilon]** (Hintzelmann, Joachimoglu, and Ohle). 292, **Oxidation of anethole** (Horiuchi and Uyeda). 298, **Isomeric terpineols; Active terpineols from active pinenes; Isomeric carvomenthols from carvacrol** (Paolini); **Caryophyllene series** (Henderson, Robertson, and Kerr). 299, **Oxidation of sesquiterpenes** (Gibson, Robertson, and Sword); **Azulene** (Ruzicka and Rudolph).

Patents.

Manufacture of styrol and its homologues. Naugatuck Chemical Co., Assees. of I. Ostromislensky and M. G. Shepard (E.P. 232,909, 15.9.24. Conv., 7.5.24).—See U.S.P. 1,541,175; B., 1925, 692. The following temperatures are specified for the hydrocarbons named: *o*-methylethylbenzene 580—620°, *p*-methylethylbenzene 640—650°, 1:3:5-dimethylethylbenzene 640—650°, *p*-cymene 650°. The hydrocarbon vapour is passed through a heated conduit under such conditions that if R be the rate of flow in g. per min., T the temperature, and V the volume of the conduit in c.c., then the value of $K=(0{\cdot}064V/R+T)/633$ falls within the limits 0·8 to 1·2.

Producing lead compounds [lead tetraethyl]. B. Mead, Assr. to General Motors Corp. (U.S.P. 1,567,159, 29.12.25. Appl., 14.6.23).—An electrolytic cell for producing lead tetraethyl comprises a porous pot surrounding an annular lead cathode, the anodes being a number of graphite rods outside the pot. The catholyte is an emulsion of 10 pts. of ethyl iodide in 100 pts. of 10% sodium hydroxide, containing 1 pt. of casein. The anolyte consists of 10% sodium hydroxide. The porous pot is provided with a reflux condenser and an agitator and the cell is heated to 55°. On passing a current of 1·15 amp. per sq. dm. lead diethyl is formed in the porous pot and decomposes into lead tetraethyl and lead. The lead tetraethyl is separated and distilled in steam. The process gives good yields and can be applied to the preparation of other lead alkyls.

T. S. Wheeler.

Obtaining absolute propyl alcohol. J. A. Steffens, Assr. to U.S. Industrial Alcohol Co. (U.S.P. 1,568,349, 5.1.26. Appl., 5.4.21).—Aqueous *iso*propyl alcohol containing 12% of water is mixed with a quantity of benzene equal to the alcohol and is passed to a steam-heated rectifying column from the bottom of which anhydrous *iso*propyl alcohol is recovered whilst a ternary mixture containing 19% of *iso*propyl alcohol, 8% of water, and 73% of benzene passes to the top of the column, whence a portion is returned to form a reflux, whilst the remainder is condensed; when cool it forms two layers, the upper containing benzene 79%, water 3%, and *iso*propyl alcohol 18%, and the lower water 73·5%, benzene 0·5%, and *iso*propyl alcohol 26%. The upper layer rich in benzene is passed to the column and the lower layer is led to a second column where it is rectified so that vapours containing 80% of *iso*propyl alcohol pass from the top of the column and are condensed and returned to the first column, whilst water flows from the bottom of the second column. The process can be applied to *n*-propyl alcohol.

T. S. Wheeler.

Preparation of solutions of mercurated hydroxybenzenesulphonic acids and their homologues. Chemosan A.-G. (G.P. 419,609, 30.9.24. Conv., 23.9.24. Addn. to 399,394).—Solutions of therapeutic value, containing the mercury derivatives in molecular dispersion, are produced by neutralising a gel of the mercurated compound, using phenolphthalein or an indicator having the same range of colour change; or an accurately known weight of the gel is brought into solution, and two equivalents of sodium hydroxide solution are added. The process renders unnecessary the addition of neutral salts, as in the process described in the chief patent (cf. B. 1924, 927). For example, mercurated 2-hydroxy-5-sulphobenzene-1-carboxylic acid gel is heated below 70—75°, with sufficient water to yield a 1·5% solution. After addition of two equivalents of sodium hydroxide, the solution can be evaporated until it contains 5—6% Hg.

L. A. Coles.

Preparation of a Peru balsam substitute. O. Schmatolla (G.P. 419,732, 7.10.24).—A product, similar to Peru balsam but possessing superior antiseptic powers and ease of manipulation, is obtained by dissolving condensation products of benzoic and cresotic acid derivatives in benzyl benzoate or benzyl alcohol. Thus, powdered sodium cresotate is slowly added to a cold mixture of benzoyl chloride, benzene, and alcohol, the temperature being maintained at 35°. Calcium carbonate is added and after keeping for 24 hrs., the mixture is warmed to 60°, with addition of alcohol and benzene, and filtered. The solvents are slowly distilled off, and by further heating of the residue for some hours at 60° a soft resin is obtained which is only slightly volatile in the cold and is soluble in benzyl benzoate and benzyl alcohol.

E. H. Sharples.

Preparation of colloidal metal solutions. A. Binz (G.P. 417,973, 14.2.23).—The solutions, which are of use in chemotherapeutics and as plant sprays, are obtained by treating metal salts with reducing agents in the presence of condensation products obtained by the action of formaldehyde-sulphoxylates upon arsenobenzene derivatives substituted with amino-groups, such as 3:3′-diamino-4:4′-dihydroxyarsenobenzene, or upon arylarsinic acids, such as 3-amino-4-hydroxybenzene-1-arsinic acid. For example, a solution of sodium hyposulphite in 0·1*N*-sodium hydroxide is added to a mixture of aqueous solutions of 3:3′-diamino-4:4′-dihydroxyarsenobenzenemethylenesulphoxylate and copper sulphate, yielding after a short time a deep red solution of colloidal copper from which a reversible copper

colloid is precipitated by the addition of acetone. Solutions containing colloidal silver or mercury can be prepared by the same method, using sodium hyposulphite or hydrazine hydrate as reducing agent.
L. A. COLES.

Solid protected colloids in soluble form. H. VOGEL (G.P. 422,150, 12.10.22).—The colloid is precipitated by a second colloid with which it will form a peptisable insoluble mass which can be purified by washing etc. As an example, silver protected by glutin can be precipitated with tannic acid and peptised, after kneading with water to remove electrolytes, by addition of a small quantity of a base.
E. S. KREIS.

Separating butyl alcohol and water. A. STEVENS (Reissue 16,267, 16.2.26, of U.S.P. 1,394,232, 18.10.21. Appl., 9.10.24).—See B., 1921, 877 A.

Purifying alcohols and oils (G.P. 422,313).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Influence of desensitisers on the coloured tints of photographic papers. J. MILBAUER and J. LAUSCHMANN (Chem. Listy, 1926, 20, 12—17). —Addition of small quantities of Phenosafranine and Pinakryptol Green to the usual developers prevents the formation of warm tones, such as light brown or blood-red, during the development of silver chloride papers. The resulting positives are black or brownish-black. The rate of development increases appreciably with only 0·001% of desensitiser. With very dilute solutions of the developer coloured tones may be obtained even in the presence of the desensitiser, except in the case of amidol. Spent developing solutions or those diluted so that they produce only light brown tones will produce satisfactory black to brownish-black images, if the above desensitisers are added in suitable proportion (cf. B., 1925, 693).
A. R. POWELL.

Photochemical oxidation of leuco-bases. CARROLL.—See A., Mar., 253.

PATENTS.

Photographic gelatin emulsion surfaces. J. LEYDE, Assee. of E. LEYDE (E.P. 241,152, 19.2.25. Conv., 9.10.24).—Gelatin emulsion surfaces are coloured with a light-filtering dye by treatment with an alcoholic solution of a water-soluble dye (*e.g.*, for the orange-red, a mixture of Acid Aniline Red and Tartrazine, and for the green, a mixture of Tartrazine and Patent Blue, Höchst) to which has been added a quantity of water such that, by the time complete drying has occurred, the dye will have penetrated into the gelatin for a small distance which is essentially less than the thickness of the surface changed by the action of light. The method may be applied to the production of a film band provided with alternately differently coloured screen-sections by pressing absorbent rollers dipping into the dye solution against the emulsion surface which is covered with a movable stencil so that alternate bands of the film surface may be exposed.
A. W. HOTHERSALL.

Films for instantaneous *X*-ray exposures. P. SPECKLIN (F.P. 591,629, 8.1.25).—The body of the film contains some substance which, under the influence of *X*-rays, emits radiations which intensify the action of the *X*-rays. Suitable substances are calcium tungstate, barium platinocyanide, zinc sulphide, quinine sulphate, Eosin, and metallic zinc, copper, nickel, iron, etc. The exposure time is shortened considerably.
W. CLARK.

Positive blue-print paper. DÜRENER FABR. PRÄPARIERTER PAPIERE RENKER UND CO. (G.P. 419,177, 28.3.25. Addn. to 403,902; cf. B., 1925, 229).—Salts of heavy metals, suitable ones being those of copper, molybdenum, and uranium, which are reduced by light are used, and subsequently treated with ferricyanide or ferrocyanide to give a coloured compound.
W. CLARK.

Photographic developer for development in bright light. F. HILDEBRANDT (G.P. 419,179, 23.11.24).—To a concentrated developer solution is added a concentrated solution of desensitiser, together with a colloid, *e.g.*, dextrin, gum arabic, or, especially, water-glass, to prevent precipitation of the desensitiser.
W. CLARK.

Selenium toning bath for photographic developing and printing papers containing silver. LEONAR-WERKE ARNDT UND LÖWENGARD (G.P. 419,428, 17.7.24).—A selenium toning bath appreciably stronger than other known baths is prepared by dissolving selenium by boiling in an aqueous solution of sodium carbonate with addition of hyposulphite.
W. CLARK.

Development of images from diazo-compounds and components capable of coupling. KALLE UND CO. A.-G. (G.P. 419,987, 4.12.23). —Development is effected in an enclosed space in which ammonia gas is evolved from substances yielding it. The process is simple and cheap.
W. CLARK.

Toning silver images. K. PETERS (G.P. 421,011, 6.11.24).—In addition to salts of light and heavy metals, the toning bath contains compounds formed by the action of nitrites on hyposulphites in feebly acid solution. Under these conditions salts of molybdenum, tungsten, nickel, and cobalt act like gold salts.
W. CLARK.

Desilvering old [photographic] films. W. TRAXL (Austr. P. 100,855, 8.4.24).—The desilvering bath consists of 3—6% nitric acid containing about 1% of manganous sulphate, 1% of copper sulphate, or, preferably, 0·1—0·2% of potassium dichromate. The silver is then precipitated from this solution as chloride.
W. CLARK.

Photographic media and process. M. C. BEEBE and A. MURRAY, Assrs. to WADSWORTH

Watch Case Co. (U.S.P. 1,574,357, 23.2.26. Appl., 31.8.22).—See E.P. 203,285; B., 1924, 493.

Infra-red sensitisers (U.S.P. 1,568,667).—See IV.

XXIII.—SANITATION; WATER PURIFICATION.

Fundamentals in the conditioning of boiler water. R. E. Hall (Proc. Eng. Soc. W. Pa., 1925, **41**, 347—390).—The conditioning of boiler water requires more than the softening of the water, since the questions of corrosion and scale formation on the heating and evaporating surfaces, and the entrainment of moisture and sludges and the presence of non-condensable gases in the steam must be considered. The scale in boilers is due to the deposition of crystals *in situ* from a saturated solution; the adherent deposits from natural waters consist chiefly of calcium sulphate and hydrous magnesium silicate, whilst a softer porous scale of calcium carbonate is often formed in the feed water lines or the preheater. The addition of soda-ash to the feed water will prevent the formation of adherent scale, the calcium being precipitated as carbonate and the magnesium as hydroxide, which can be removed easily. At high pressures or high ratings, however, soda-ash reacts with the water with the evolution of carbon dioxide; under these conditions sodium phosphate is used. The sulphate/carbonate or sulphate/phosphate ratio of the water must be very carefully controlled and depends on the nature of the water and the operating pressure of the boiler. Corrosion in the feed-water lines or the boiler is prevented by de-aeration of the water to remove dissolved oxygen, and by controlling the hydrogen-ion concentration by the addition of caustic soda. The amount of solid matter or sludge in suspension in the boiler will depend on whether the treatment of the water is external or internal and the proportion of condensed water from turbines, process work, etc. used to feed the boiler. The steam pipes must be protected by mechanical means from moisture and suspended solids entrained by the steam, so that the liability of corrosion and erosion by the condensing steam is minimised. It is very difficult to deal with the non-condensable gases in the steam (oxygen dissolved in the feed water, hydrogen by interaction of steam and the metal of the boiler, and carbon dioxide from the decomposition of soda-ash). If the presence of carbon dioxide in the steam is objectionable, sodium phosphate must be used for conditioning the water in place of soda-ash. In the discussion, the difference between water softening and boiler water conditioning was emphasised. It was pointed out that the removal of the sludge in the boiler is a mechanical question, involving external filtration and periodical blow-downs. The prevention of scale deposition generally leads to a tendency to form wet steam. B. W. Clarke.

Presence of air in pure and in alkaline water. J. Porter (J. Roy. Tech. Coll. Glasgow, 1925, [2], 19—25).—When pure water is heated to 100° it still retains about 11 c.c. of air per litre and this is removed only by prolonged boiling. Addition of 4 g. of sodium hydroxide per litre increases the rate of evolution of air at temperatures above 60° and the air retained at 100° is only 4·8 c.c. per litre. The solubility of air in 4% sodium hydroxide solution at 17° is 8 c.c. per litre, compared with 20·4 c.c. in pure water. Very little air is evolved on heating water until a temperature above 80° is reached, and experiments are described which indicate that the air which is not evolved as it ought to be below 80° forms a layer of no appreciable volume on the sides of the vessel and is not retained in supersaturated solution. If the water is maintained for a proplonged period at a temperature of 60°, however, all the excess air over the normal amount that will remain in solution at that temperature, is slowly liberated. Addition of a slight amount of oil to water during heating causes a more regular evolution of the dissolved air.
A. R. Powell.

Chlorophenol tastes in Milwaukee's water supply. R. W. Cunliffe (J. Amer. Water Works Assoc., 1925, **14**, 423—433).—The chlorinated water supply of Milwaukee, taken from Lake Michigan, on occasions in 1915 developed a taste which appeared to be the same or very similar to the taste of a dilute solution of chlorine. In 1917 and 1918, the taste occurred more frequently and was intense; whereas previously it had occurred during winter months, and was attributable to retarded absorption and dissipation of the chlorine on account of low temperatures, in 1918 the tastes occurred in summer and were persistent and marked. The taste was not affected by boiling, and residual chlorine was present in less amount during periods of taste than normally. Experimental filtration of various types, aeration, carbonation, and treatment with thiosulphate and other chemicals failed to have any effect. Samples of liquid trade waste from the different industries in the city were collected, chlorinated, boiled to remove traces of free chlorine, cooled, and tasted. Only wastes from gas plants and industries the effluents from which contained products associated with the destructive distillation of coal were found to possess taste, and of the products from this source only phenol and cresol in great dilution produced taste of stability and flavour comparable with those which had been experienced. Phenol in dilutions greater than 1 to 200,000 pts. of water could not be detected by taste, but after addition of a fraction of 1 p.p.m. of chlorine could be detected in dilutions of 1 pt. of phenol in 250—500 million pts. of water. That the tastes complained of were due to chlorophenols was confirmed by keeping the sources of pollution of the lake water by phenolic liquors under observation. The principal source of the trouble was waste liquor, discharged direct into the lake, derived from a phenol manufacturing plant; in some cases effluents from Semet-Solvay coke-oven and coal gas plants were responsible. Wastes from these sources, with one exception, are now disposed of by mixing with sanitary sewage.
W. T. Lockett.

Differential media for detection of *B. coli* in water. H. G. DUNHAM, M. H. MCCRADY, and H. E. JORDAN (J. Amer. Water Works' Assoc., 1925, 14, 535—549).—A statement of the progress made to date in the work of the American Standardisation Council Committee (Standard Methods of Water Analysis), having reference to the present Amer. Publ. Health Assoc. standard method for determining the presence of the colon group of lactose-fermenting aerobes. Investigations include a study from a cultural standpoint of the dyes of American manufacture available for use in eosin-methylene-blue-agar, brilliant-green-bile medium, and gentian-violet-lactose broth; a comparative study of brilliant-green-lactose-bile (ox gall) and standard lactose broth; and an enquiry as to the value of brilliant-green-lactose-bile (ox gall) as a confirmatory medium. The results obtained by a large number of independent workers indicate that brilliant-green-lactose-bile is not entirely satisfactory for use as a medium for the *B. coli* presumptive test, a lack of sensitiveness as compared with standard lactose broth being noticed with some waters. The medium, however, apparently eliminates practically all spore-bearing lactose fermenters. Investigations carried out at Indianapolis regarding the value of brilliant-green-lactose-bile as a confirmatory medium, indicate that the interpolation of this medium, following growth in lactose broth, would result in the elimination of a large number of Endo-plates. Apparently, when used in this manner, brilliant-green-lactose-bile does not inhibit any organisms of the colon group, but the concentrations of brilliant-green (1:10,000) and bile (5%) used do not seem to be such as to be absolutely specific for colon group organisms. Gentian-violet-lactose-bile as confirmatory medium behaved similarly to brilliant-green-lactose-bile (cf. Howard and Thompson, B., 1925, 735). W. T. LOCKETT.

Prophylaxis in industrial lead poisoning. D. MCKAIL (J. Ind. Hyg., 1926, 8, 74—77). A prescription is given for a mouth wash and drink so designed as to cause precipitation of lead as sulphate. H. J. CHANNON.

Cause of "gulf" disease. P. BIGINELLI (Giorn. Chim. Ind. Appl., 1925, 7, 568—571).—This disease, which has appeared on the shores of the Gulf of Dantzig, is caused by toxic gases formed by putrefaction of waste liquors from (1) cellulose factories in which the sulphurous acid used is prepared from arsenical pyrites and (2) the treatment of arsenical hides. T. H. POPE.

See also pages 260, **Hazards from use of "ethyl-gasoline"** (FLINN). 264, **Effect of dyes used in paper-making on animal life in water** (HAEMPEL). 265, **Poisoning by chloronitrobenzene and acetanilide** (RENSHAW and ASHCROFT). 272, **Solid calcium cyanide** (Metzger). 281, **Volatilisation of lead during lead burning** (ENGEL and FROBOESE).

PATENTS.

Softening of water. F. SCHMIDT (E.P. 227,429, 18.12.24. Conv., 12.1.24).—Caustic alkalis and carbonates, borates, phosphates, and like salts of alkali metals are used in conjunction with suitable alkali compounds of acids insoluble in water (silicates, aluminates, aluminosilicates) to form a water-softener. The softening medium may be made into a solid or plastic mass by means of clay, mucilaginous substances, casein, and the like. D. G. HEWER.

Manure from sewage and other substances. F. STÖTZEL (E.P. 224,226, 27.10.24. Conv., 3.11.23). —Sewage is filtered through a bed of peat, peat litter, or the like, and the resulting mass of sewage solids and filtering material is mixed with unburnt gypsum and/or kainite in a finely-ground condition to form a dry mass. Industrial refuse, animal waste substances, and/or chemical substances may then be added, and, if required, the mass, with or without further addition of nitrogenous substances, is pulverised. W. T. LOCKETT.

Sewage purification plant. M. PRÜSS (E.P. 235,872, 12.6.25. Conv., 19.6.24).—The plant consists of a combined settling basin and digestion chamber separated by thin metal walls. The settling basin has a narrow and deep channel at the bottom, which communicates by means of a drag-pipe with an outer gutter at the top end of the digestion chamber. The levels of the water in the settling and digestion chambers are so arranged that the sludge from the bottom of the former can be conveyed up the pipe into the gutter by a siphon action and can be distributed as required to different parts of the digestion chamber. C. T. GIMINGHAM.

Preparation of sterilising agents, germicides, and the like. C. H. H. HAROLD (E.P. 245,820, 7.10 and 15.12.24).—A sterilising liquid is prepared, in which the active agent is mono- or di-chloroamine or a mixture of the two, by adding the correct proportion of chlorine in concentrated solution or as gas to an aqueous solution of ammonia containing not more than 0·0125 g. per litre. Under these conditions, the yield of chloroamines is almost quantitative. If the chlorine is allowed to diffuse through a porous diaphragm, and the ammonia solution is caused to circulate, thus avoiding local rise of temperature due to the heat of the reaction, the strength of the ammonia solution may be increased up to 0·75 g. per litre. C. T. GIMINGHAM.

Preheating and purifying [deaerating] boiler feed-water. C. HÜLSMEYER (E.P. 245,588, 14.7.24).

Degasifying or deaerating liquids [boiler feed water]. W. S. ELLIOTT (E.P. 247,342, 18.12.24).

Bacterial filters for purification of sewage and other liquids. C. R. B. BROWN (E.P. 247,769, 27.3.25).

Hydrogen-ion concentration of waters (G.P. 399,410 and 400,273).—See XVI.

Mixtures containing finely-divided copper compounds (G.P. 419,460).—See XVI.

Insecticides etc. (E.P. 246,252).—See XVI.

Insecticide (U.S.P. 1,568,044).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 30, 1926.

I.—GENERAL; PLANT; MACHINERY.

Influence of exterior temperature on the temperature of standard pyrometric [electric] lamps. G. Ribaud (Compt. rend., 1926, 182, 625—627).—The influence of the external temperature on the apparent temperature of a tungsten filament in a standard pyrometric lamp is investigated theoretically under conditions of constant current, constant resistance, and constant voltage. Short, thick filaments require appreciable corrections under the first-named condition, this being confirmed experimentally. S. K. Tweedy.

Trichromatic colorimeter suitable for standardisation work. J. Guild (Trans. Optical Soc., 1925-6, 27, 106—129).—A trichromatic colorimeter which may be employed to obtain the specification of any colour in a form which can be converted to any fundamental basis of specification is described. The three working primary colours are obtained by means of gelatin filters, and mixing of these colours is effected by a periscopic prism which rotates past three stationary sectors. Provision is made for adding any of the primaries to the colour under test, should this be necessary in order to obtain a match of colours, so that colours of higher saturation than it is possible to match directly may be dealt with. J. S. G. Thomas.

Criticism of the monochromatic-plus-white method of colorimetry. J. Guild (Trans. Optical Soc., 1925-6, 27, 130—138).—It is contended that from the practical standpoint of quantitative colorimetry, hue and saturation are not fundamental elements of colour quality, but must be regarded as derivatives of the trichromatic constitution of the colour. It is unsound to determine colour quality by methods involving a photometric measurement. Such determinations should involve only those properties of the eye which are concerned in colour-match relationships. J. S. G. Thomas.

New method of colorimetry. J. Guild (Trans. Optical Soc., 1925-6, 27, 139—158).—A colorimeter, employing a new method of determining the quality of a colour, as defined by its position on the trichromatic colour chart, in which only the colour-matching properties of the eye are involved, is described. The determination depends on two colour matches in which the test colour is matched by a mixture, in unknown proportions, of a standard colour and a monochromatic colour produced spectroscopically, the standard colour being different in the two matches. J. S. G. Thomas.

Laboratory mixing machine for solids.—R. M. Hixon (Ind. Eng. Chem., 1926, 18, 138).—Small quantities of powders, *e.g.*, nicotine dusts, or solids and liquids may be mixed by means of an apparatus consisting of a stirring motor to the shaft of which is attached by means of suction tubing a metal rod covered with rubber tubing and sufficiently long to take the rotating mixing bottle. Parallel with this rod is another rod which is rigidly supported from the same stand and is provided with a glass or metal sleeve to rotate with the bottle. D. G. Hewer.

Source of error in mechanical analysis of sediments by continuous weighing. J. R. H. Coutts and E. M. Crowther (Trans. Faraday Soc., 1925, 21, 374—380).—See B., 1926, 31.

Silica to glass and to metal joints. Buttolph.—See A., Mar., 264.

Patents.

Separation of the several components of a gaseous mixture. G. L. E. Patart (E.P. 232,986, 27.4.25. Conv., 26.4.24).—An elaborate succession of columns and heat interchangers for the separation of several constituents of a gaseous mixture by liquefaction and rectification is described. For example, a pure mixture of hydrogen and nitrogen is obtained from illuminating gas (subjected if desired to catalytic hydrogenation after addition of water-gas) by a reflux stream of liquid nitrogen, which is liquefied in another column the condenser of which is cooled by liquid methane. The column for methane is cooled by liquid ethylene, and the ethylene column by liquid ammonia. B. M. Venables.

Low-temperature cooling, liquefaction, and separation of not easily condensable gases. A. Seligmann (U.S.P. 1,573,752, 16.2.26. Appl., 3.3.24).—The greater portion of the necessary decrease of pressure is obtained by the flow of the gases at a relatively high velocity through pipes adapted to absorb the pressure energy by the friction of the gases over the entire length of their walls. H. Holmes.

Means for effecting solution of solids in liquids. Wolff u. Co., E. Czapek, and R. Weingand (E.P. 245,683, 12.8.25).—An apparatus for effecting rapid dissolution of solids in liquids consists of two containers for the liquid, connected at top and bottom. The solid is placed in one of the containers, and on dissolving forms locally a solution of greater density which sinks through the

solvent, setting up circulation between the two compartments. An adjustable valve situated in the lower connecting channel controls the rate of flow of solvent to solid, and hence the rate of solution. Modifications are described in which one container surrounds the other; by spacing the outer container but slightly from the inner the exothermic heat of solution of the substance can be utilised for preheating the solvent, thereby facilitating the process. R. B. Clarke.

Automatic means for letting off steam from a container having in it a boiling liquid. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 246,567–8, 28.10.24).—Steam is blown off from a vessel containing boiling liquid through an orifice within the steam-outlet pipe. In (a) the rate of flow of steam is held constant firstly because the pressure in the vessel is above the critical value, and when the pressure has dropped below that value an automatic valve is opened by the reduction of pressure in the vessel and permits additional steam to exhaust through a by-pass. In (b) the liquid in the vessel is prevented from frothing by proportioning the areas of the orifice, of the steam pipe, and of the exposed surface of the liquid in the vessel so that the rate of flow of steam is below the frothing limit. When the liquid is water the proportions may be 1 : 12 : 9000. B. M. Venables.

Means for running off hot liquids from a container under pressure. W. Carpmael. From Farbenfabr. vorm. Bayer & Co. (E.P. 246,569, 28.10.24).—The danger of blowing down a high-pressure boiler or similar apparatus is obviated by having a pressure-tight receiver at a lower level with both steam and water connexions to the boiler. The receiver is first filled with steam up to the boiler pressure, the blow-down (water) valve is then also opened and water from the boiler discharged by gravity into the receiver. Finally, with both connexions to the boiler closed, the pressure in the receiver is reduced by blowing off steam, and the liquid then exhausted under a moderate pressure. B. M. Venables.

Vacuum evaporating apparatus. E. C. R. Marks. From Swenson Evaporator Co. (E.P. 247,246, 13.9.24).—Evaporators of the Yaryan type are described, with all parts in contact with the liquid constructed of corrosion-resisting material, *e.g.*, iron containing 11—15% Si, or glass for the tubes and earthenware for the separators. The remainder of the apparatus is made of stronger material, such as steel. B. M. Venables.

Evaporation or inspissation of solutions. Carrier Engineering Co., Ltd., and K. J. R. Robertson (E.P. 247,346, 2.1.25).—Solidified products are obtained from liquids—particularly salt from brine—by spraying the liquid saturated at ordinary temperature into a current of air at 315—370°, whereby the solution loses 5—10% of its water and is raised in temperature to 60—77°. On cooling, 2 or 3% of the liquid crystallises in the form of large crystals, and the mother liquor is returned with fresh brine to the evaporator. B. M. Venables.

Removing dust from gases or vapours. E. M. Salerni (E.P. 247,274, 10.11.24).—The gases are passed in a zig-zag course through a number of hanging plates or slats which are struck by projections carried by a moving endless chain or band at the bottom of the apparatus. The collecting plates may be so arranged that, when struck, the plates of one row scrape the surfaces of the plates of the adjacent row, and the endless band may also serve as a conveyor to remove the dislodged dust. B. M. Venables.

Mixing granular substances and liquids. P. Lenart (E.P. 247,451, 25.6.25).—The apparatus consists of a receiving vessel, a chamber with radial blades, and a centrifugal disc arranged in order downwards. The material flung off by the disc is caught in a casing and returned through a pipe to the upper vessel. The radial blades in the intermediate chamber guide the material to the centre of the disc. B. M. Venables.

Grinding machine. A. E. Jacobson (U.S.P. 1,572,722, 9.2.26. Appl., 10.9.24).—A cylindrical casing is provided with one closed side and with an axial opening in the other side, and a rotary shaft extends axially of the casing through the closed side. Spaced discs at right angles to the shaft constitute a rotary grinding head. These discs are secured to spaced arms disposed between them and carried by a member secured to the shaft. Each disc, except that adjacent to the closed side of the casing, is provided with a central opening enabling material delivered through the axial opening to pass through the casing. The disc adjacent to the closed side and the member carrying the spaced arms form a non-perforated side of the grinding head. H. Holmes.

Drying. J. Credo, Assr. to Louisville Drying Machinery Co. (U.S.P. 1,573,144, 16.2.26. Appl., 24.5.24).—The material, during its passage through the dryer, is heated and subjected to regulated currents of heated air flowing in opposite directions. H. Holmes.

Purification, filtration, decolorisation, and deodorisation of liquids and gases. H. W. A. Branco (F.P. 598,826, 27.5.25. Conv., 27.5.24).—The liquid or gas is passed through a layer of ashes from wood, coal, or peat that have been washed to remove acidic and basic substances. A. R. Powell.

Utilisation of residues by vacuum distillation in presence of steam. G. P. Guignard (G.P. 421,787, 9.1.24. Conv., 9.11.23).—Steam or water in small quantity is fed continuously into the interior of the material being distilled *in vacuo*. The material is constantly stirred, the steam or water being introduced through the hollow vanes of the stirring apparatus. A. B. Manning.

Rotary furnace. A. H. Pehrson (U.S.P. 1,574,932, 2.3.26. Appl., 8.8.22).—See E.P. 184,810; B., 1924, 39.

Dissolving device. E. Czapek and R. Weingand (U.S.P. 1,574,561, 23.2.26. Appl., 1.9.25).—See E.P. 245,683; preceding.

Dehydrating minerals. W. R. Wade, Assr. to New York Zinc Co. (U.S.P. 1,574,950, 2.3.26. Appl., 28.3.23).—See E.P. 213,561; B., 1925, 193.

Apparatus for drying granular material. F. H. Rosencrants, Assr. to Internat. Combustion, Ltd. (U.S.P. 1,576,362, 9.3.26. Appl., 15.10.24).—See E.P. 234,327; B., 1925, 577.

Disintegrating dry powders. H. Plauson (U.S.P. 1,575,717, 9.3.26. Appl., 3.11.22).—See E.P. 195,690; B., 1923, 534 A.

Pulverising machine. R. S. Riley, Assr. to Sanford Riley Stoker Co. (U.S.P. 1,576,472, 9.3.26. Appl., 21.7.24).—See E.P. 236,769; B., 1925, 746.

Evaporator for concentrating or drying. T. Rigby (U.S.P. 1,576,471, 9.3.26. Appl., 23.11.21).—See E.P. 181,035; B., 1922, 574 A.

Separation of larger from smaller grains of granular substances. F. H. Clause (E.P. 248,082, 28.11.24).

Combined powdered-fuel and grate furnaces. C. Hold (E.P. 248,277, 19.9.25).

Shaft furnaces for burning limestone etc. (E.P. 245,653).—See VII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Bacterial decomposition of textile fibres. III. Occurrence of humus compounds in deteriorated fabrics and the bearing of their formation on the origin of peat and coal. A. C. Thaysen, W. E. Bakes, and H. J. Bunker (Biochem. J., 1926, 20, 210—216).—Microbiological activity is insufficient to eliminate all the cellulose present in plant tissues in the process of peat formation by decay. Humic compounds obtained from typical peats consist of two different types, one of which yields a chloro-derivative identical with or very similar to that of "natural humus" (lignin humic compound), and the other a chloro-derivative closely related to that of artificial humus compounds obtained by the action of inorganic acids on carbohydrates or from cellulose fibres decayed through ageing. It is suggested that the presence of this latter humus compound in peat may be due to the decomposition of cellulose other than by microbiological activity. A humus compound of this type was isolated from a sample of Egyptian linen dating from the 18th Dynasty. S. S. Zilva.

Chemistry of coal. IV. Oxidation of the residue from the benzene-pressure-extraction process. W. A. Bone and R. Quarendon (Proc. Roy. Soc., 1926, A 110, 537—542; cf. B., 1924, 584).—When the residue obtained after extraction of a bituminous coal with benzene under pressure was treated with alkaline potassium permanganate, complete oxidation and solution resulted (cf. Francis and Wheeler, A., 1925, i., 1389). About 25—40% of the residue taken was recovered in the form of acid oxidation products from which considerable yields of mellitic and benzene-1 : 2 : 3 : 4-tetracarboxylic acids have been obtained. Other unidentified acids are being investigated. The large yield of mellitic acid indicates that the coal substance has a six-carbon ring structure, each carbon of the ring being connected to other carbon atoms. F. G. Soper.

Coking of coal. H. Greger (Braunkohlenarch., 1925, 67—101; Chem. Zentr., 1926, I., 1904—1905).—The softening of coal during coking was studied by measuring the pressure necessary to force a wire loop a fixed depth per unit of time, into compressed blocks of the powdered coal. The degree of softening first decreases and then increases with rising temperature. The softening curves and the maximum softening vary with different varieties of coal. For any particular variety the softening curve and the appearance of the coke are affected by preheating the coal, the preheating temperature and the duration of the heating both having an effect. The softening and swelling properties of the preheated coal are depreciated by storing in the absence of air after preheating. L. A. Coles.

Ash from powdered-fuel installations. J. T. Dunn (J.S.C.I., 1926, 45, 60—61 T).—Ashes escaping into the air from powdered-fuel installations have been impossible to obtain, but the author has measured the size of particles in ashes from the flues and boiler tubes in such installations, both in Britain and in the United States. The fineness of these particles ran from 1/120 in. to 1/6000 in., and over 85% of the whole were under 1/800 in. The particles escaping are presumably still finer, and the rate of settlement of these from the air must be so slow that they will be spread over an enormous area, and cause no nuisance by their settlement in the neighbourhood of the installations producing them. This accords with the fact that deposits of ash have not been obtained in the neighbourhood of powdered-fuel installations.

Resistance to the flow of gases in the fuel bed of a coke-fed furnace or water-gas generator. M. W. Travers (J.S.C.I., 1926, 45, 61—63 T).—The author discusses the results of the experiments carried out by the U.S. Bureau of Mines (Tech. Paper 137), with a view of calling attention to the need for further investigation of the subject. In shallow fuel beds the fact that the pressure in the ash-pit increases as the 1·5th, and not as the 2nd. power of the rate of flow of the air into the fuel bed, is probably due to the fact that the spacing of the material of the fuel bed is dependent upon the blast velocity. The resistance to the flow of the air

is the sum of two factors, the loss of head due to the increases in velocity and in mass of the gas, which attain a maximum in the hottest part of the fuel bed, and a factor which is proportional to the depth of the fuel bed, and which represents the loss of head due to viscosity.

Determination of phenols in crude ammonia liquor at coking plants and gasworks. F. Ulrich and K. Kather (Z. angew. Chem., 1926, **39**, 229—232).—Koppeschaar's method of determining phenols by the bromine they take up is applicable to mixtures of phenol and the isomeric cresols, provided the mean mol. wt. of the mixture is known. Three atoms of bromine are taken up per mol. of the phenol. Other substances reacting with bromine (*e.g.*, hydrogen sulphide, ammonium thiosulphate) must first be eliminated, and this may be done by extracting the ammonia liquor with benzene containing 20% of quinoline. The method takes 2 hrs., and gives results in good agreement with those obtained by the gravimetric, ether extraction method.

W. T. K. Braunholtz.

[U.S.] Bureau of Mines Orsat apparatus for gas analysis. A. C. Fieldner, G. W. Jones, and W. F. Holbrook (U.S. Bur. Mines Tech. Paper No. 320, 1925. Pp. 18).—Carbon dioxide is determined by absorption in caustic alkali; unsaturated hydrocarbons by absorption in fuming sulphuric acid (with subsequent removal of the sulphur trioxide fumes in the caustic alkali); oxygen by absorption in alkaline pyrogallol solution; carbon monoxide by combustion over copper oxide heated at 300° by an electric furnace (or provision may be made for the determination of carbon monoxide alone by absorption in ammoniacal cuprous chloride). By adding oxygen or air and burning in a combustion pipette, methane and ethane may be determined. The apparatus is fitted with a compensating tube for eliminating errors due to changes in pressure, temperature, and pressure of aqueous vapour. The apparatus is somewhat similar in principle and method of operation to Haldane's gas burette and some of its modifications, although it is arranged like Orsat's. The compensator, however, is a separate connexion, not being attached to the caustic alkali absorption pipette. It is essential that the copper oxide tube shall be cool when, after having been used, it is being rinsed during the methane and ethane determination. Before commencing an analysis the free spaces in the apparatus must be filled with nitrogen. Sources of error and their avoidance are indicated and a table of pertinent data relating to the commoner gases is given.

R. A. A. Taylor.

Karwendel oil shale. E. Berl and W. Schmid (Brennstoff-Chem., 1926, 7, 49—54).—Karwendel shale yields an oil of high sulphur content which is the source of products of pharmaceutical value (ichthyol). Analysis of the shale gave: moisture 1·25%, volatile matter 37·98%, sulphur 3·98%, nitrogen 0·36%, ash 52·08%. On distillation under ordinary pressure in an iron retort holding 600 g., to a maximum temperature of 545°, the shale yielded 13·4—14·4% of oil and 3·8—4·6% of gas, whilst at pressures of 10—50 mm. the yields were 17·9—18·8% of oil and 4·9—5·5% of gas. The average composition of the gas was: CO_2 14·13%, H_2S 23·71%, N_2 3·28%, H_2 19·31%, CH_4 31·34%, CO 2·15%, O_2 0·86%, C_nH_m 4·95%. A thick, red-brown oil with green fluorescence, unpleasant odour, and feebly alkaline reaction was obtained; d^{18} 0·975, S 9·98%. Fractionation under 12—20 mm. pressure gave 35—120° 26·54% (S 12·71%), 120—220° 35·77% (S 11·12%), 220—280° 30·07% (S 10·45%), and pitch 7·62%. The oils darkened rapidly on keeping, due to oxidation of the unsaturated constituents. The phenol content of the oil was less than 1%. Only a very small amount of the sulphur was removed by treatment with copper oxide. The crude oil was purified by treating successively with soda-lime, sodium and ammonia, dilute acid, and finally distilling under ordinary pressure from 120° to 230°. The oil thereby lost its unpleasant odour. The purified oil gave the characteristic colour reactions of dimethylthiophen. Treated with liquid sulphur dioxide at −12°, the purified oil left 8·12% undissolved. This fraction, containing the saturated hydrocarbons, was bright yellow, odourless, and contained S 1·96%. The soluble fraction, containing the unsaturated and aromatic hydrocarbons, was Bordeaux red in colour after removal of the sulphur dioxide, possessed a pleasant odour, and had a sulphur content of 8·61%. The calorific value of the dry shale was 3713 cal./g.; the calorific values of the oil fractions and gases have been estimated from their composition.

A. B. Manning.

Oil-bearing chalk at Heide (in Holstein). G. Keppeler and J. Schmidt (Z. angew. Chem., 1926, **39**, 220—224).—Benzene extracts from the chalk 10—20% of a heavy, black, viscous, and highly unsaturated oil, which is similar to, though not identical with, that exuded by the chalk *in situ.* Attempts to extract the oil by dissolving away the chalk with acid were unsuccessful. Distillation of the chalk alone yields about 75% of oil, 15% of gas, and 10% of dark, asphaltic residue (reckoned on the total oil content of the chalk), whilst distillation in a current of steam, carbon dioxide, or hydrogen yields about 89% of oil and 5—6% of gas. The oils obtained by distillation are lighter than the natural and extracted oils, and, unlike the latter, can be distilled without decomposition, giving about 2—6% of benzine, 45—50% of oil distilling at 150—350°, and 45—50% of lubricating oils. Oil obtained by distillation in a current of gas has a higher sulphur content than that obtained by direct distillation, and that obtained by distillation in hydrogen has an exceptionally low flash point and low content of unsaturated aliphatic hydrocarbons.

W. T. K. Braunholtz.

Conversion of fatty acids into hydrocarbons. G. Stadnikov and E. Ivanovski (Neftjanoe i slancevoe Chozjajstvo, 1925, 8, 470—476; Chem. Zentr., 1926, I., 1909).—On passing 940 g. of fatty

acids from linseed oil having saponif. value 188, and iodine value 179, over finely divided iron deposited upon asbestos, at 400°, a gas mixture containing mainly carbon dioxide with a little carbon monoxide and ethylene hydrocarbons, and 720 g. of liquid distillate, were obtained. The fraction of the distillate volatile in steam (b.p. 50—250°) was hydrogenated and then dehydrogenated over nickel, to convert hydroaromatic into aromatic hydrocarbons. The liquid, however, consisted almost entirely of aliphatic hydrocarbons. On treating with potassium permanganate the portion volatile in steam (iodine value 139), the greater part was oxidised to carbon dioxide, but small quantities of the lower fatty acids, up to butyric acid and valeric acid, and succinic acid, were formed. L. A. COLES.

Naphthenic acids from gas oil distillate of Californian petroleum. Y. TANAKA and S. NAGAI (J. Soc. Chem. Ind. Japan, 1926, 29, 1—7; cf. B., 1925, 196).—From the product obtained by acidifying a waste lye produced in the refining of the gas oil fraction of the mixed oil from Long Beach and Santafe Spring Fields in California, a mixture of crude naphthenic acids, d_4^{15} 0·9797, n_D^{15} 1·4927, acid value 191·4, was separated. These were converted into the methyl esters, d_4^{15} 0·9535, n_D^{15} 1·4806, 81% of which boiled at 160—230°/8·9—9·0 mm. By hydrolysis of the esters pure mixed naphthenic acids were obtained, having d_4^{15} 0·9773, n_D^{15} 1·4893, neutralisation value 208·4. They distilled at 136—260°/8·9—9·0 mm., the chief portion (82%) distilling at 200—260° and having d 0·976—0·985; the fraction distilling at 210—220° had d 0·985. The specific gravity (0·976—0·985) of the main fraction is a little lower than that (0·99) of the naphthenic acids derived from Kurokawa petroleum (B., 1922, 973 A) and a little higher than that (0·97) of the acids from Nishiyama petroleum (cf. B., 1925, 870). There are two series of isomeric naphthenic acids differentiated chiefly by the specific gravity, and the acids from Californian petroleum are probably a mixture of the two series of the acids. A white crystalline mass separated in the fractions of high boiling point. K. KASHIMA.

Effects of lead tetraethyl upon the deterioration of turbine oils. S. HATTA (J. Soc. Chem. Ind. Japan, 1925, 28, 1346—1352).—Quantities of 0·05, 0·1, or 0·2% of lead tetraethyl were added to turbine oils, which were then kept at 120°, and treated with moist oxygen passed at the rate of about 150 bubbles per min. With an oil of the paraffin series, the darkening of the colour, sludge formation, and the deterioration of demulsifying power were distinctly diminished by the presence of lead tetraethyl. The density, viscosity, and acid and saponification values of the oil, however, increased gradually, both in the presence and absence of the lead compound. With an oil of the naphthenic series, the darkening of the colour was retarded, but the density, acid and saponification values, and the sludge formation were all distinctly increased by the presence of lead tetraethyl. K. KASHIMA.

Micrographic analysis of mineral oils subjected to oxidising processes. M. VANGHELOVITCH (Bul. Soc. Chim. Romania, 1926, 7, 93—99; cf. HOBLYN, B., 1925, 274).—The work of Hoblyn (*loc. cit.*) is extended. Samples of oil were heated at 250° for varying periods and examined microscopically, the time required for the first precipitation of asphalt being noted. This test gives a much more reliable indication of the value of the oil than the older tests of viscosity, inflammability, covering power, etc. Chemical analysis of the oil after heating gives results agreeing with the microscopical method, but the latter is much quicker and more delicate for detecting the first changes. By the use of various solvents on the precipitates it is shown that the oxidation proceeds in the order: oil→hard asphalt →soft asphalt→carbene (colloidal carbon)→coke. The long periods of heating may be avoided by using nitric acid. A thin film of oil is placed under the microscope and a drop of a solution of nitric acid added. If the concentration of the acid exceeds a certain limit an immediate precipitation of asphalt occurs, and by noting the limiting concentration of nitric acid required by different oils, the resistance of the oils to oxidation is obtained. Results thus obtained agree with those found by prolonged heating at 250°. W. HUME-ROTHERY.

Adsorption of resins by paraffin wax and solidification of paraffin-containing products. N. TSCHERNOSHUKOV (Neftjanoe i slancevoe Chozjajstvo, 1925, 8, 640—646; Chem. Zentr., 1926, I., 1910—1911).—On removal of resin from masut of maximum and minimum solidifying pts. 25° and —2°, these values being recorded after heating to 60° and 100° respectively (cf. Tytschinin, B., 1924, 1003), the solidifying point rose to 25—27° and was independent of previous heating; on removing the greater part of the ozokerite, it fell to 2—15°, and was dependent upon previous heating; on removing both resins and high-molecular paraffins, it was 14° and was independent of previous heating. Colorimetric investigation of the adsorption of resins by kerosene saturated with paraffin wax, showed that adsorption increases with rising melting point of the wax, and the quantity of resin adsorbed at different temperatures varies according to the solidifying point of the product. L. A. COLES.

Liquid reaction products obtained by action of hydrogen on paraffin wax under high pressure at 450°. H. I. WATERMAN and A. F. H. BLAAUW (Rec. trav. chim., 1926, 45, 284—295; cf. B., 1925, 273, 746).—Careful fractionation of the gasoline boiling up to 150°, obtained by the action of hydrogen at 450° and pressures up to 280—290 atm. on Rangoon paraffin wax containing C 86·4%, H 14·8%, indicated that the gasoline contains large quantities of the successive members of the paraffin series (n-pentane to n-nonane) and probably a smaller amount of olefines (about 10%). Benzene was not detected and toluene is only present to a very small extent. R. BRIGHTMAN.

Effect of heat on mineral oils. Products of decomposition of the oil by the electric arc.

F. Evers (Wiss. Veröff. Siemens-Konz., 1925, 4, 324—334).—When a high-tension arc is maintained under mineral oil the gaseous products are hydrogen (50·3%), methane (1·0%), nitrogen (3·4%), oxygen (0·9%), and unsaturated hydrocarbons (44·4%), of which the main portion is acetylene, with a little ethylene. These unsaturated products were separated as bromides and from the less volatile fraction a *bromide*, $C_6H_4Br_8$, m.p. 181°, was isolated. Distillation experiments on the oil prove that some liquid products also result from the action of the arc. A dark brown solid which is produced is of variable composition but contains oxygen (4—7%), hydrogen (3%), and carbon (90—94%). The colloidal properties of this solid and the manner of its production are discussed. G. M. Bennett.

Determination of ammonia in ammoniacal liquor. Jungblut.—See VII.

Insulating oils. Stäger.—See XI.

Patents.

Drying wet fuels. E. Berl (G.P. 419,906, 6.11.23).—A liquid, insoluble or very sparingly soluble in water, is intimately mixed with the fuel, and the excess liquid then removed by centrifuging or pressing. The process is repeated several times. The oils obtained by the distillation of lignites or bituminous coals are particularly suitable for the purpose; other liquids which can be used are mineral oils, aniline, toluidine, phenols, etc. A. B. Manning.

Preparation of low-ash coal. Elektro-Osmose A.-G. (Graf Schwerin Ges.) (Swiss P. 100,129, 18.1.22. Conv., 29.1.21).—A suitable electrolyte, *e.g.*, an alkali silicate, is added to the finely divided material, which is then subjected to a flotation process. A. B. Manning.

Briquettes from powdered fuels. E. Kollár (Swiss P. 97,593, 9.7.21. Conv., 14.7.20).—The powdered fuel is mixed with a pulp made by heating finely divided non-lignified parts of plants (leaves, stems, etc.) with water at 60—100°. To make the briquettes waterproof, plant materials containing tannin and resins may be incorporated with the pulp, or the formed briquettes may be dried in flue gases. A. B. Manning.

Peat briquettes. J. Steinert (Swiss P. 99,230, 9.8.21. Conv., 28.1.21).—Peat is dried in the field to a water content of 45—50%, further artificially dried to 12—15% water content by direct treatment with hot gases of low oxygen content, and then made into briquettes. The gases for drying are supplied by a producer fed with peat of about 30% water content. A. B. Manning.

Manufacture and use of active carbons. J. N. A. Sauer (E.P. 247,241, 13.8.24).—A base of active carbon is impregnated with an organic or inorganic substance such as will by suitable subsequent treatment form an adsorbent (*e.g.*, silica gel, activated carbon) in the pores of, or on, the basis. The apparent sp. gr. of the active carbon is thus increased and also the adsorptive power per unit volume. The process may be used to effect an agglomeration of the carbon particles to give larger granules. R. A. A. Taylor.

Producing highly adsorbent charcoal. E. Bateman, Assr. to United States of America (U.S.P. 1,573,509, 16.2.26. Appl., 5.8.19).—Carbon in a comminuted form is treated with carbon dioxide at 600—1000°. R. A. A. Taylor.

Carbon from coal, lignite, peat, wood, and waste material containing these substances. J. G. Aarts (F.P. 598,023, 4.3.25. Conv., 14.1.25).—The material is distilled and the gaseous products are decomposed in the usual way to give amorphous carbon. A. R. Powell.

Distillation of carbonaceous materials. E. M. Salerni (E.P. 247,300. Appl., 14.11.24).—The apparatus comprises a drying chamber fitted with plates or shelves and a carbonising chamber. In the dryer the charge is scraped by means of bars on an endless chain so that it passes along and over the edge of one plate and falls on to the plate below. On the return journey the bars scrape it along and off the edge of the second plate. Finally it passes through a gas-tight valve into the carbonising chamber. This may be of the same type as the dryer, or it may consist of a series of troughs arranged side by side, the charge passing laterally through them in succession. The carbonised residue may then be passed into a cooler of similar construction to the dryer. R. A. A. Taylor.

Distillation of lignites. E. Heneage (E.P. 247,324, 28.11.24).—Several horizontal metal cylinders are arranged one above the other and the charge is passed through one by means of a screw-conveyor and then drops into the next cylinder below. The charge is dried in the topmost and carbonised in the lower cylinders, after which it passes out of the setting into a cooling cylinder. The flues are arranged for heating the setting from the bottom, but the screw-conveyors may have hollow shafts to admit of the introduction of hot air or gases into the cylinders to heat the charge by their sensible heat. The screw-conveyors are flanged in order to take up heat from the cylinder walls and pass it to the charge. R. A. A. Taylor.

Distillation of coal and other fuels. "Allkog" Allgem. Kohlensverwertungs-G.m.b.H. (F.P. 598,568, 22.5.25. Conv., 16.7.24).—The coal or fuel is mixed with sand or other inert material, before distillation, in order to improve the heat transference, and to loosen the fuel. A. B. Manning.

Production of a low-boiling tar by the carbonisation of lignite. Chem. Fabr. Griesheim-Elektron, Assees. of P. Siedler and K. H. Küster (G.P. 421,617, 14.2.22).—The charge of coal is heated in cylindrical, vertical or inclined retorts, and the temperature is raised so slowly and the heating carried out so regularly, that at the beginning of the

actual tar evolution the coal is sufficiently free from water. The heating is slowly continued, avoiding the formation of layers at different temperatures. The products are led off at the lower end of the retort. The tar so obtained is distinguished from the lignite tars previously described in that 95% or more distils below 200°, and it is, moreover, more or less free from phenols. A. B. MANNING.

Distillation of coal. GES. FÜR INDUSTRIE-OFENBAU M.B.H. (G.P. 423,125, 21.12.17).—Coal is so distilled that the tar is obtained from it at a lower temperature, the gas and ammonia at an intermediate temperature, and the coke at a higher temperature. The distillation is carried out by the passage through the coal of gases which are heated first by the sensible heat of the discharged coke, then by the nearly completely coked charge, which is externally heated. A. B. MANNING.

Continuous water-gas production. H. HILLEBRAND (G.P. 422,999, 28.1.22).—The necessary heat of reaction for water-gas production is supplied by a stream of gas which circulates through a heater and then through the producer. The sensible heat of the gas stream after leaving the producer is used to evaporate the water for the water-gas production. Before reheating the gas stream any excess water it contains is separated. The process permits easy regulation of the gas composition. A. B. MANNING.

Pre-drying and pre-distilling gas-producer fuels by hot gases. F. LANDSBERG (G.P. 423,350, 30.4.19).—A small part of the gas from the drying zone is led with the fuel into the carbonising zone, while the remainder passes away above. Separation of the drying layer from the carbonising layer by a valve is not necessary. A. B. MANNING.

Continuous production of water-gas and producer-gas. A.-G. FÜR INDUSTRIEWERTE, and J. FRÜH (Swiss P. 97,229, 5.3.21).—A gas containing oxygen acts on finely divided coal in a rotary retort heated internally by electrical means. A. B. MANNING.

Production of oil-gas. GES. FÜR CHEM. IND. IN BASEL (Swiss P. 98,743, 1.7.22).—By cracking gas-oil or gas-oil vapour in a current of oil-gas itself a considerable increase in the yield of carbon in the form of gaseous hydrocarbons is obtained. A. B. MANNING.

Production of combustible gases from finely divided or high-ash fuels. STETTINER CHAMOTTE-FABR. A.-G. VORM. DIDIER (Swiss P. 98,993, 17.12.21. Conv., 21.12.20).—Low-grade fuel is fed into a retort, ignited, and heated by passing air through the charge in such a manner that a hot porous mass of clinker is formed over the cross-section of the retort above the air port. The air, passing through this, comes into contact with the unburnt fuel in a highly heated and finely divided state. A. B. MANNING.

Combustible gas mixture containing nitrogen. N. F. O. PALACIN (Swiss P. 112,359, 21.2.25).—A portion of a mixture of carbon monoxide and nitrogen obtained by burning coal over a mixture of calcium carbonate with coal, is caused to react with steam, yielding, after removal of carbon dioxide, a mixture of nitrogen and hydrogen, which is then mixed with the remainder of the original gas. L. A. COLES.

Determination of the constituents of gas mixtures. ADOS G.M.B.H., and K. HENSEN (G.P. 423,142, 12.11.24).—The gas mixture is sent in succession through two capillary tubes maintained at different constant temperatures, and the pressure in a vessel lying between the two tubes is observed. The pressure varies with changing viscosity of the gas mixture. The temperatures of the capillaries are chosen to give large pressure differences, which are recorded by an attached manometer. The readings are not affected by changes in the external temperature. A. B. MANNING.

Apparatus for the examination of gas mixtures, especially flue gases. ADOS G.M.B.H., and K. HENSEN (G.P. 423,297, 30.8.24).—A shaft rotating at constant speed carries two propellers, one of which rotates in the gas mixture under examination, and the other in a comparison gas. The shaft can move axially under the resultant force of the two propellers, which act in opposition to one another. The axial motion of the shaft automatically alters the inclination of the blades of the second propeller in such a way that the force due to the propeller in the gas mixture is increasingly opposed until equilibrium is reached. A pointer attached to the shaft registers its axial displacement. A. B. MANNING.

Iron carbonyl mixtures [for use as motor fuel]. BADISCHE ANILIN- & SODA-FABR. (F.P. 597,517, 1.5.25. Conv., 5.12.24).—The mixtures contain hydrocarbons and at least 10% of iron carbonyl, a stabiliser (1 : 4-di-*p*-toluidinoanthraquinone) and dyes being added if necessary.

Distillation and fractional condensation of complex mixtures, such as mixed hydrocarbons. SOC. ANON. D'OUGRÉE-MARIHAYE (F.P. 598,414, 8.9.24).—The mixture is subjected to a circulatory process, in which the mixed vapours traverse the lighter condensation products in the lower, middle, and upper zones successively, of a condensing column. In the middle zone definite pure products are obtained. A. B. MANNING.

Manufacture of a highly-active decolorising charcoal. H. MÜLLER-CLEMM, Assr. to GES. FÜR CHEM. PRODUKTION M.B.H. (U.S.P. 1,575,703, 9.3.26. Appl., 2.5.24).—See E.P. 216,130; B., 1925, 490.

Apparatus for removing solid carbonaceous material from oil-cracking apparatus. L. C. HUFF (E.P. 248,232, 2.6.25).—See U.S.P. 1,528,004; B., 1925, 749.

Separation of gaseous mixtures (E.P. 232,986).—See I.

Utilising energy liberated during oxidation of hydrogen compounds of sulphur, nitrogen, etc. (G.P. 421,665).—See VII.

III.—TAR AND TAR PRODUCTS.

Production of road tars without distillation. L. H. SENSICLE (Gas World, 1926, 84, Coking Sect., 31—36).—In the coke-oven plant where the method was devised, the gases passed through a cyclone tar extractor working at 95°, followed by a centrifugal extractor at 85°. Liquor was sprayed into the latter. The light oil was largely carried forward and condensed previous to benzol washing. In order to prepare road-tar, a suitable tar mixture, from which part of the light oil had been removed, was passed through a preheating boiler and into a de Laval separator at 55—60°. This reduced the water content to 1% and further dehydration was effected by air agitation in the tar receiver, a temperature of 60° being maintained. Free carbon from the tar gradually accumulated in the bowl of the separator and required removal. C. IRWIN.

Working up lignite low-temperature tar without distillation. II. R. VON WALTHER, H. STEINBRECHER, and W. BIELENBERG (Braunkohlenarch., 1925, 59—63; Chem. Zentr., 1926, I., 1908—1909).—The influence of distillation in a current of steam and *in vacuo* upon the characteristics of low-temperature tar has been studied. However carefully the process be carried out, the character of the tar is altered considerably during distillation.
L. A. COLES.

Removing creosote from tars and tar-oils by means of solid calcium hydroxide. R. VON WALTHER and W. BIELENBERG (Braunkohlenarch., 1925, 64—66; Chem. Zentr., 1926, I., 1906).—Considerable quantities of creosote are removed from tars and tar-oils by treatment with solid calcium hydroxide, but a small proportion always remains, the proportion varying with different tars. The use of petroleum ether or benzine as solvent for the tar during the treatment gave favourable results, but when benzol or ether was used, only moderate quantities of creosote were extracted.
L. A. COLES.

Obtaining useful products by the oxidation under pressure of lignite-tar creosote. E. KÁRPÁTI (Chem. Runds. Mitteleuropa u. Balkan, 1926, **3**, 3—5; Chem. Zentr., 1926, I., 1908).—Oxidation under pressure is used for purifying phenolic fractions of b.p. up to about 230°, and for converting liquid tar products containing a high proportion of creosote into solid condensation products having good electrical insulating properties.
L. A. COLES.

PATENTS.

Distillation of tar. B. YOUNG (G.P. 408,945, 12.12.22).—Tar is distilled in a rotary retort, the interior of which is provided with a scraping device, which removes the pitch and coke formed during the process. Tar of high water and dust content can be continuously distilled in the apparatus.
A. B. MANNING.

Working up of tar. H. SUIDA (Austr. P. 101,333, 19.10.23).—Treatment of the tar takes place in two stages; in the first the tar is distilled to liquid pitch, and in the second the pitch is distilled to coke. The coke is gasified by the introduction of air into the retort, the gas produced being used for heating the distillation plant. The pitch obtained in the first stage is led continuously into one of two generator-retorts, in which alternately the pitch is coked and the coke is gasified. A. B. MANNING.

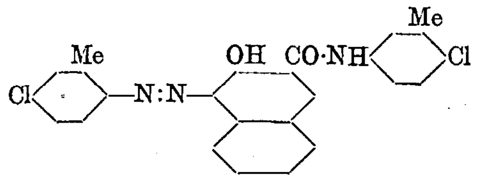
Fractional condensation of hydrocarbons (F.P. 598,414).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Constitution of Hansa Yellow G [M.L.B.] and other yellow pigment colours. F. M. ROWE, A. H. BURR, and S. G. CORBISHLEY (J. Soc. Dyers and Col., 1926, **42**, 80—82).—Hansa Yellow G, $NHPh{\cdot}CO{\cdot}CHAc{\cdot}N_2{\cdot}C_6H_3Me{\cdot}NO_2$, was prepared by coupling diazotised *m*-nitro-*p*-toluidine with acetoacetanilide. The latter could only be obtained in 30% yields by heating ethyl acetoacetate with aniline at 130—140° under pressure; at ordinary pressures, using an inert solvent, diphenylcarbamide was the chief product. The methods of E.P. 9252 of 1914 and G.P. 287,569 and 293,429 (B., 1915, 867) gave satisfactory yields. Monolite Yellow G (B.D.C.) and Pigment Fast Yellow HGL conc. new (J.W.L.) are identical with Hansa Yellow G (M.L.B.). Pigment Fast Yellow GRL Extra (J.W.L.) was made by coupling diazotised *m*-nitro-*p*-toluidine with acetoacet-toluidide. The toluidide (unlike the anilide) is obtained in satisfactory yields by heating equimolecular proportions of ethyl acetoacetate and *o*-toluidine at 150° under pressure. Pigment Fast Yellow RL Extra (J.W.L.) is identical with the GRL brand but is shaded with a small proportion of an insoluble azo colour. It has similar properties to, but is not identical with, Hansa Yellow R (M.L.B.). Helio Fast Yellow RL (By.) appears to be a mixture of the dibenzoyl derivatives of 1 : 5- and 1 : 8-diaminoanthraquinones.
A. COULTHARD.

Composition of some products used for the production of insoluble azo colours. F. M. ROWE and E. LEVIN (J. Soc. Dyers and Col., 1924, **42**, 82—93; cf. B., 1925, 797).—Naphthol AS-TR (Gr.E.), which is used in conjunction with diazotised Fast Red TR base (Gr.E.) and Fast Scarlet TR base (Gr.E.) is the 5-chloro-*o*-toluidide of β-hydroxynaphthoic acid. It has been prepared by condensing equimolecular proportions of the components in boiling toluene in the presence of phospho ru trichloride. Fast Red TR base is the hydrochloride of 5-chloro-*o*-toluidine. The colour produced on the fibre by using it in conjunction with Naphthol

AS-TR (Gr.E., G.P. Appl. 48,892) is the compound described by Saunders (J. Soc. Dyers and Col., 1924, 48) as of outstanding fastness to kier boiling, viz.,



Fast Scarlet TR base (Gr.E.) is the hydrochloride of 6-chloro-*o*-toluidine. Diazotised and coupled with β-naphthol it gave red needles, m.p. 166°, and with Naphthol AS, red needles with metallic lustre, m.p. 268°. Naphthol AS-D (Gr.E.), which is also used in conjunction with the above bases, is the *o*-toluidide of hydroxynaphthoic acid (E.P. 23,732 of 1913, 199,771, G.P. 293,897; B., 1914, 70; 1923, 824 A). The following colouring matters have been prepared (cf. B., 1924, 704): Diazotised Fast Red TR base, coupled with Naphthol AS-TR, gives scarlet needles, m.p. 285°, with Naphthol AS-D, red needles, m.p. 261°. Diazotised Fast Scarlet TR base and Napthhol AS-TR gives scarlet needles, m.p. 268°, and with Naphthol AS-D, long scarlet needles, m.p. 272°.

A. COULTHARD.

See also A., Mar., 296, **Sulphonic acids of arylamine derivatives of naphthaquinones** (WAHL and LANTZ). 297, **Constitution of naphthazarin** (DIMROTH and RUCK). 307, **Transformation of 2 : 4-dinitro-4′-phenylazodiphenylamine into phenazine derivatives** (BERETTA). 314, **Potentiometric titration (diazotisation) of amines** (MÜLLER and DACHSELT).

Trichromatic colorimeter. Criticism of monochromatic-plus-white method of colorimetry. New method of colorimetry. GUILD.—See I.

PATENTS.

Manufacture of condensation products [dyes] of the anthraquinone series. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 246,683, 23.2.25. Add. to 205,502; B., 1924, 627).—The condensation products, suitable for use as dyes or for the production of dyes, described in the chief patent and in E.P. 220,304 and 222,125, are obtained more easily and in better yield by prolonged treatment of a 1 : 5-diaroylnaphthalene with aluminium chloride at a high temperature and preferably with the simultaneous introduction of air or oxygen into the reaction mixture. According to the temperature at which the operation is conducted and to its duration dibenzopyrenequinones are obtained directly, or *Bz*-1-aroylbenzanthrones or their reduction products are first formed, these being capable of subsequent conversion into dibenzopyrenequinones. For example, a dye having m.p. about 385° and soluble in nitrobenzene is obtained by heating, within a closed vessel, for 2 hr. at 170—195° a mixture containing 1 pt. of 1 : 5-dibenzoylnaphthalene, m.p. 186·5°, and 10 pts. of freshly sublimed aluminium chloride or alternatively by heating for 40 hr. at 120° and with the continuous introduction of oxygen, a mixture containing 29 pts. of benzanthrone, 190 pts. of aluminium chloride, and 116 pts. of benzoyl chloride, the temperature being further maintained at 180° for 24 hr.

A. J. HALL.

Manufacture of sulphide dyes. A. G. BLOXAM. From A.-G. FÜR ANILIN-FABR. (E.P. 247,378, 10.2.25).—Sulphide dyes of good fastness on cotton are obtained by melting sulphur, or a compound which yields sulphur (other than strongly alkaline sulphurising agents such as alkali sulphides or polysulphides), with leuco-indophenols in which the hydrogen of the amino-group has been substituted by the methylene or benzylidene group, the benzyl group, an acyl group of the aliphatic or aromatic series, or a substitution derivative of these groups which has a tendency to form a thiazole ring during sulphiding. For example, 15 pts. by weight of 4-amino-4′-hydroxydiphenylamine are added to 10 pts. of benzaldehyde. The solid benzylidene-compound produced is baked with 60 pts. of sulphur for 10 hr. at 200°. The crude melt is dissolved in aqueous sodium disulphide and the dye precipitated by a current of air. Cotton is dyed from an alkali sulphide bath of the dye in reddish-brown shades of very good fastness to soap, boiling, acid, and chlorine.

A. COULTHARD.

Manufacture of dyes of the dibenzanthrone series. BADISCHE ANILIN- & SODA-FABR. (F.P. 598,752, 25.5.25. Conv., 11.8.24).—Aminodibenzanthrones when treated with acylating substances under mild conditions yield derivatives which, in the presence of diluents, may be alkylated or aralkylated. For example, benzoyl chloride in nitrobenzene acts on aminodibenzanthrone to form a vat dye giving a blue shade on cotton which changes to grey in the presence of chlorine. Amino*iso*dibenzanthrone gives a violet dye. The dye formed by the action of *o*-chlorobenzoyl chloride on aminodibenzanthrone when treated with methyl toluenesulphonate gives a blue vat dye, fast to chlorine. Aminobenzanthrone and toluenesulphonyl chloride in nitrobenzene yield a dye which gives a greenish-blue shade on cotton and is converted by methyl toluenesulphonate into another dye (crystalline) giving a similar shade.

A. COULTHARD.

Manufacture of trisazo dyes. FARBENFABR. VORM. F. BAYER & Co. (F.P. 598,894, 28.5.25. Conv., 13.6.24).—Diazo compounds of primary disazo dyes which have a 1-amino-2-naphthyl ether 6-sulphonic acid as end component are coupled with 2-amino-5-naphthol-7-sulphonic acid or 2-arylamino-5-naphthol-7-sulphonic acid or their derivatives, in the presence of pyridine or its derivatives. The dyes so produced, give pure greenish-blue shades on cotton, which are fast to light. For example, the diazo compound of the disazo dye from aniline-3 : 6-disulphonic acid, α-naphthylamine, and 1-amino-2-naphthyl ether-6-sulphonic acid is coupled with

2-phenylamino-5-naphthol-7-sulphonic acid.
A. COULTHARD.

Preparation of polyazo dyes. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of L. LASKA and F. WEBER (G.P. 423,092, 16.11.24).—One mol. of a tetraazo compound—from benzidine for example—is combined with 1 : 8-aminonaphthol-3 : 6-disulphonic acid, in a solution of mineral acid, and the product coupled with a monodiazo-compound and 1 : 8- or 1 : 5-naphthylenediamine. The dyes are specially suited for mixed fabrics of wool and cotton.
A. COULTHARD.

Production of hydroxyanthraquinones. J. THOMAS, H. W. HEREWARD, and SCOTTISH DYES, LTD. (E.P. 246,529, 22.10.24).—In the manufacture of hydroxyanthraquinones, particularly alizarin, by treating 2-chloroanthraquinone with caustic soda and an oxidising agent in an autoclave, as described in E.P. 174,101 (B., 1922, 212A), the resulting alizarin is insoluble in 20—25% caustic soda and the presence of sodium chloride in the reaction mixture has no harmful effect. A simplified and more economical method of working up the autoclave melt is thus possible, the alizarin being separated by direct filtration or filtration after formation of insoluble calcium alizarate (by addition of lime or calcium hydroxide) and the alkaline filtrate concentrated and causticised so that it may be again used although it contains sodium chloride.
A. J. HALL.

Preparation of 1 : 2 : 3 : 4-tetrahydroxyanthraquinone. FARBENFABR. VORM. F. BAYER & Co., Assees. of R. E. SCHMIDT (G.P. 421,235, 7.2.24).—Alizarin in concentrated sulphuric acid, is treated with suitable oxidising agents, such as manganese dioxide, lead peroxide, etc., in the presence of boric acid. The process runs smoothly and no purpurin is formed.
A. COULTHARD.

Preparation of a heterocyclic compound of the naphthalene series. GES. FÜR CHEM. IND. IN BASEL (Swiss P. 100,851, 2.12.22. Addn. to 92,688; cf. A., 1923, i., 938).—*α-Napthathiofuran-1 : 2-dione*, a red crystalline powder, m.p. 168°, is obtained by the action of oxalyl chloride upon *α*-thionaphthol, preferably in the presence of diluents, *e.g.*, carbon disulphide or benzene, and condensing agents, *e.g.*, aluminium chloride or sulphuric acid.
L. A. COLES.

Dyestuffs of the anthraquinone series. J. BADDILEY and W. W. TATUM, Assrs. to BRITISH DYESTUFFS CORP. (U.S.P. 1,574,748, 2.3.26. Appl., 18.4.23).—See E.P. 201,610; B., 1923, 968A.

Waste waters from dyeworks (Austr. P. 100,735).—See XXIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Influence of sunlight on wool. W. VON BERGEN (Textilber., 1925, 6, 745—751; Chem. Zentr., 1926, I., 1070).—After exposure to sunlight wool swells strongly in dilute alkali and crumples characteristically; it shows no double refraction between crossed nicols. Acids promote the decomposition of the wool molecule, and the oxidation of the liberated sulphur to sulphuric acid. Alkalis hinder the oxidation of the sulphur, and neutralise the acids formed. Free sulphuric acid causes yellowing of the wool. After treatment with sunlight wool is dyed more deeply by basic dyestuffs, and less so by acid dyestuffs.
A. GEAKE.

Behaviour of textile fibres towards colloidal solutions [of soap, Turkey-red oil, etc.]. W. HERBIG and H. SEYFERTH (Z. Deuts. Oel- u. Fett-Ind., 1926, 46, 81—84).—The wetting properties of aqueous solutions containing soap, Turkey-red oil, Turkey-red oil and Tetracarnit (Sandoz), Bäuchol (a proprietary wetting-out substance), Bäuchol and Tetracarnit, and Tetracarnit were compared by measurement of (1) the degree and rapidity with which the solutions (tinted with Benzopurpurine 4B) ascend vertically suspended cotton and woollen yarns, (2) their surface tension and viscosity by means of a stalagmometer and Ostwald's viscosimeter respectively, (3) the degree of spreading of drops of the solutions along a single horizontal cotton thread, and (4) the vertical depth of drops of the solutions suspended from a horizontal band of cotton threads. The results obtained are inconclusive and depend on factors (*e.g.*, the tension and surface of the textile fibres) other than the wetting-out properties of the solutions.
A. J. HALL.

Importance of a definite regulation of atmospheric humidity in industrial textile processes. J. OBERMILLER (Z. angew. Chem., 1926, 39, 46—51; cf. B., 1925, 94).—For spinning and weaving processes it is important to maintain a definite and comparatively high relative humidity of the surrounding air; the strict regulation of the temperature is less vital. Curves are drawn showing the relation at 20° between the relative atmospheric humidity and the moisture content of wool, raw silk, fibroin, artificial (viscose) silk, and cotton. These are all very similar and are roughly linear between about 10% and 75% relative humidity. At a given relative atmospheric humidity the moisture content of the various fibres shows but slight variation with temperature. Cotton, a vegetable fibre which contracts when wetted, shows an increased elasticity in the wet state, whereas the reverse is true for animal fibres, such as wool and silk, and also artificial silks, which expand when wetted.
W. T. K. BRAUNHOLTZ.

Chemistry of the sulphite-cellulose digestion process. E. Hägglund (Svensk Kem. Tidskr., 1925, 37, 116—124; Chem. Zentr., 1925, 96, I., 271; cf. Hägglund and Bjorkman, B., 1925, 238, 586).—In the early stages of the digestion of wood with solutions containing sulphur dioxide and sulphites, one or more ligninsulphonic acids are formed which cannot be removed from the pulp by washing with water. The lignin in wood is chemically combined with carbohydrates, and the sulphonic acids of these compounds hinder penetration of the sulphite liquor. During digestion, the sulphite liquor reacts rapidly

at first and then but slowly. Accumulation of ligninsulphonic acids within the pulp occurs if the rate of hydrolysis of the lignin–carbohydrate compounds is slower than the rate of sulphonation, but this increase is hindered by a rise in the temperature of digestion. Sufficient lime should be present so that the digestion is complete or nearly complete before combination of all the sulphite occurs. Ligninsulphonic acid is comparable in strength with mineral acids. During digestion the concentration of ligninsulphonic acids approaches the original concentration of the sulphur dioxide. At any moment during digestion, the lime present is distributed between the ligninsulphonic acids and the sulphite, and at the completion of digestion the greater part is combined with the former. The digestion liquor remains bright yellow provided that sufficient sulphite is used, and darkening is caused by the use of high temperatures. Blackening of the cellulose occurs when the acidity of the digestion liquor increases too much by formation of ligninsulphonic and sulphuric acids, insufficient lime being present. A. J. Hall.

Chemical activity of cellulose and its significance in sizing paper. E. Oeman (Papier-Fabr., 1925, 23, 725—727, 745—748, 800—802, 813—816).—Cellulose is amphoteric in character; thus, if immersed in dilute acid, hydrogen ions are taken up and hydroxyl ions are formed, and the converse takes place in an alkaline medium. The isoelectric point is somewhat on the acid side of neutrality. When sulphite-cellulose is steeped in 0·015*N*-sulphuric acid, washed thoroughly, and immersed in water, the amount of alkali required to bring the p_H of this water to 8 is several times as great as when the cellulose has not been previously immersed in acid. This is not due to retention of sulphuric acid because no sulphate can be found in the alkaline solution, and it is ascribed to combination of cellulose with hydrogen ions; this shifts the isoelectric point towards the acid side. With sulphate-cellulose the effect is smaller, and immersion in alkali has the reverse effect. The sulphate ions, corresponding to the hydrions taken up from sulphuric acid, remain in the solution as free ions. Thus, if a trace of sulphuric acid is added to water, and this shaken with sulphate-cellulose, the p_H of the water, determined electrometrically with a hydrogen or quinhydrone electrode, is less than that determined colorimetrically. If the water is made alkaline the electrometric method gives the higher p_H. Size particles are electro-negative and, by making the cellulose positive by absorption of hydrogen ions from an acid solution, combination can be made to take place without alum, but this is not a practical process. When alum is used aluminium is absorbed and the cellulose becomes electro-positive; this is enhanced by washing with acid water, and size then readily combines and is fixed in the fibre.
A. Geake.

Theory of resin sizing. E. Oeman (Papier-Fabr., 1926, 24, 49—58).—Sulphite-cellulose takes up aluminium from alum solutions and calcium passes into the solution. The amount of aluminium which is absorbed depends on the calcium content of the cellulose, and becomes a minimum for ash-free filter paper. Aluminium is not absorbed as hydroxide, but probably replaces calcium in some organic combination. If cellulose containing aluminium or calcium is washed with water, some of the metal passes into solution, calcium more freely than aluminium. If more sulphite-cellulose is added to the wash water the aluminium is absorbed, and may thus be transferred from one portion of cellulose to another. Sulphite-cellulose, and even ash-free filter paper, are able to absorb and fix resin without any previous treatment with alum. The amount absorbed depends on the degree of dispersion of the resin sol. Thus from a sol prepared by pouring an alcoholic solution into water, sulphite-cellulose was able to absorb 55·5% of the resin; whereas from sols prepared by adding the calculated amounts of dilute sulphuric acid to sodium resinate solutions only 16—25% could be absorbed. After treatment with alum and washing, ash-free filter paper was able to absorb practically all the resin from the first sol, but only 15% from the second. Sulphite-cellulose after alum treatment was able to absorb as much as 20% of its weight of resin from a sol prepared from the alcoholic solution, although the excess of alum had been washed off and could not have passed into the resin sol. From sols prepared from sodium resinate only about 15% was absorbed. The amount of resin taken up by sulphite-cellulose after alum treatment may be increased by adding acids or neutral salts, which diminish the dispersion of the resin, but if too much is added poor sizing may result. Any alum passing into the resin solution assists sizing in this way. A. Geake.

Comparative study of paper fillers. M. B. Shaw and G. W. Bicking (U.S. Bur. Standards Tech. Papers, 1925, 19, [301], 733—748).—In continuation of previous work (cf. B., 1925, 126) experiments have been conducted on a semi-commercial scale in order to compare the paper-making qualities of a number of fillers including asbestine, talc, clays, "crown filler" (artificially prepared calcium sulphate), and gypsum. All the fillers used were of relatively good colour and low grit content. Retention figures for the silicate fillers were fairly regular, talc being the best in this respect; "crown filler" and gypsum, on the other hand, owing to their solubility in water, showed poor retentive qualities. An examination of the various samples of paper made under strictly comparable conditions showed that, in general, differences in opacity, finish, strength, and degree of sizing were due in the main to the difference in retention of the fillers. Talc, however, appeared to cause less reduction in tensile strength and bursting strain than did other fillers when present to the same extent. None of the fillers had any appreciable effect on the colour of the paper, but any outstanding differences in the colour of various fillers would be noted in the finished sheet. It is concluded that asbestine and talc are, with respect to their paper-making qualities, comparable with the better clays:

also, that relative cost is no criterion of paper-making value; the cheapest clay proved to be as good as one costing twice as much, and "crown filler" offered no advantage over the cheaper gypsum.

D. J. NORMAN.

Analysis of resin size. M. GOTTLÖBER (Papier-Fabr., 1926, 24, 125).—"Total resin" is determined by precipitating with dilute sulphuric acid, collecting the precipitate on a tared filter, drying at 105°, and weighing. Filtering the precipitate has the advantage over extracting it with ether that substances, such as casein and starch, which contribute to the sizing effect, are included. A. GEAKE.

See also A., Mar., 312, **Silk fibroin** (BRILL). 325, **Fermentation of cellulose by thermophilic bacteria** (VILJOEN, FRED, and PETERSON).

Occurrence of humus compounds in deteriorated fabrics. THAYSEN, BAKES, and BUNKER.—See II.

PATENTS.

Process for working up animal fibres, hairy skins, or hair. R. BACH (E.P. 243,301, 22.12.24. Conv., 20.11.24).—Aldehydes, ketones, or compounds capable of yielding them, more particularly bisulphite compounds, are used, either alone or in conjunction with other agents, *e.g.*, mercury salts, as carroting agents for hair and the like. Formaldehyde is especially suitable and may be used in aqueous solution or in gaseous form. This process may be applied before or during the felting operation, or to "half-planked felt (Labraz)": alternatively, these carroting agents may be added to the planking liquor. The carroting operation is preferably followed by treatment with oxidising agents; for this purpose hydrogen peroxide and nitric acid are particularly suitable, since, apart from their function as deodorisers, they enhance the carroting effect. *Example:* Hair is exposed to the action of a 5—15% solution of formaldehyde (slightly acidified with, for instance, hydrochloric acid) at 25—80° for ½—40 hr. depending on the quality of the hair and the temperature and concentration of the solution. If desired, the operation may be conducted under pressure at temperatures above 100°. The hair is then centrifuged and treated with hydrogen peroxide. This process improves the felting properties of the fibres and enables inferior and waste hair to be successfully used in the manufacture of hat bodies.

D. J. NORMAN.

Proofing cellulosic, animal, and other substances against insects and the like. S. W. KENDALL (E.P. 247,242, 14.8.24).—Cellulosic, vegetable, animal, metallic, and building materials are protected from attack by all forms of animal or vegetable organisms by impregnating or covering with an insoluble soap of a rare-earth metal, such as cerium, lanthanum, didymium, or yttrium, or of thorium, thallium, titanium, zirconium, or uranium. The soap may be applied as a true or colloidal solution in an organic solvent, or as an emulsion in water, or it may be prepared in or upon the material from an oxide, hydroxide, or salt of the metal and the organic acid or a soluble salt of the latter. Fatty, oleic, linoleic, linolenic, clupanodonic, abietic, cocceric, ricinoleic, dihydroxystearic, japanic, chaulmoogric, resin or gum acids may be used, or crude mixtures of these, such as oils, fats, waxes, gums, or resins, or chemical derivatives, such as oxidation, reduction, hydration, or dehydration products. Textile materials may be treated with such a proportion of the preparation that they are proofed against organisms without substantially affecting their appearance or ability to absorb moisture; a larger proportion also waterproofs the material (cf. U.S.P. 1,536,254; B., 1925, 586). Timber is similarly protected from dry rot, the death watch beetle, and from marine animals or vegetation; plaster, boards and the like are protected from ants and rodents. The compositions may also be used as anti-fouling paints for timber and metal. A. GEAKE.

Material for protection [of fabrics etc.] against moths. W. SCHMITZ (G.P. 421,100, 18.7.24. Addn. to 419,463; cf. E.P. 230,203, B., 1925, 421).—The material consists of a mixture of quillaia bark and lupin or broom seeds which is extracted with a dilute inorganic acid (not sulphuric acid), and the extract neutralised with sodium or magnesium carbonate, evaporated to a thick syrup, mixed with anhydrous sodium sulphate, and reduced to a dry powder. C. T. GIMINGHAM.

Manufacture of artificial filaments, films, plates, and the like. H. HAWLIK (E.P. 242,240, 7.9.25. Conv., 29.10.24).—Filaments, films, and the like, which are impervious to water, are prepared from viscose by mixing it with a base, basic compound or salt, and adding an organic acid to the coagulating bath. The addition to the viscose may be sodium aluminate, aluminium chloride, sulphate, or acetate, or other compounds of aluminium, calcium, barium, strontium, tin, or zinc, or soap, or mixtures of salts. The organic acid may be a higher fatty acid, such as palmitic, stearic, oleic, or erucic acid, a sulphonated acid, such as sulphoricinoleic acid, a hydrogenated fatty acid, hydroxy-fatty acid, halogenated fatty acid, resin acid, wax acid, or the like, or an aromatic acid, such as salicylic or tannic acid. If soap has been added to the viscose, the bath may contain a compound, such as aluminium sulphate or calcium chloride, which forms insoluble soaps. A. GEAKE.

Manufacture of artificial silk and the like from viscose solutions. W. P. DREAPER (E.P. 245,815, 17.9.24).—The drying of freshly-precipitated viscose filaments under tension prior to further treatment may be dispensed with, and the desulphurising, bleaching, and dyeing operations conducted with the filament in the gel condition if the desulphurising solution contains an alkali or ammonium salt in quantity equivalent to at least 40% saturation, preferably about 80% saturation, at the ordinary temperature. When dyeing is carried out from sodium sulphide solution, desulphurisation and dyeing may take place simultaneously provided that the dyestuff is not salted out. It is advantageous to add

an alkali salt to any other baths, *e.g.*, dyeing or bleaching, through which the filament is passed, but in such cases about 10% of the saturation quantity is sufficient. The treated and washed filaments are subsequently dried under tension on stretching frames. Yarn produced by this process behaves like raw or "gum" natural silk in that the filaments show a certain tendency to cohere.

D. J. Norman.

Manufacture of artificial silk or the like. J. C. Hartogs (E.P. 246,423, 9.11.25).—Considerably larger quantities of soaps (added either as potash soaps or suitable fatty acids) may be incorporated with viscose solution if potassium hydroxide is used instead of sodium hydroxide in the preparation and solution of the cellulose xanthate. For instance, a suitable spinning solution is obtained by adding 2% of potassium oleate (dry weight) or 1% of oleic acid to potassium-viscose. The spinning bath may optionally contain a proportion of potassium sulphate.

D. J. Norman.

Dissolving and gelatinising cellulose esters such as nitrocellulose and like cellulose compounds. V. Plinatus (E.P. 246,272, 4.12.24).—The gelatinisation of cellulose esters is effected by the action, in the presence of water, with simultaneous application of heat and pressure, of mono-, di-, or tri-butyrin. For example, in one method the cellulose ester, previously dispersed in and retaining more than 18% of water, is treated with undiluted mono-, di-, or tri-butyrin and the resulting mixture kneaded and rolled under pressure. The presence of water as a diluent not only ensures uniform gelatinisation but accelerates the process. D. J. Norman.

Production of coloured cellulose plastics and solutions. Badische Anilin- & Soda-Fabr. (E.P. 247,288, 12.11.24).—Coloured cellulose plastics are produced by milling the cellulose ester and a softening or swelling agent, such as camphor, with an organic or inorganic colouring matter which is insoluble in the other constituents of the mixture. Milling is continued until, when a sample is dissolved in a liquid which is a solvent for the cellulose ester but not for the colour, no substantial separation of the colour takes place after keeping for several days or weeks. The mass then contains the dyestuff in a colloidally, or nearly colloidally, dispersed condition and, in a thin layer, it is more or less transparent. By dissolving such plastics in solvents, solutions are obtained which are suitable for producing coloured coatings on wood, glass, leather, metals, paper, fabrics, and the like. The colouring matter may be prepared in a highly dispersed condition before incorporation with the cellulose ester, and, in one example, colloidal silver is formed in the varnish itself. A. Geake.

Cellulose acetate film composition. S. J. Carroll, Assr. to Eastman Kodak Co. (U.S.P. 1,572,232, 9.2.26. Appl., 22.4.25).—Sufficient tributyrin is added to cellulose acetate to maintain flexibility after prolonged heating at 65°.

A. Geake.

Reducing the viscosity of nitrocellulose. S. E. Sheppard and L. W. Eberlin, Assrs. to Eastman Kodak Co. (U.S.P. 1,572,248—9, 9.2.26. Appl., 15.6.25).—Nitrocellulose is treated with (A) a substantially non-nitrating gaseous mixture, containing higher oxides of nitrogen, or (B) a peroxide solution. A. Geake.

Reducing the viscosity of nitrocellulose. L. E. Branchen, Assr. to Eastman Kodak Co. (U.S.P. 1,572,266, 9.2.26. Appl., 13.5.25).—Nitrocellulose is softened by treatment with a penetrant, and its viscosity reduced by separate treatment with an aqueous alkali. A. Geake.

Preparing and preserving fibres for pulp making. E. C. Lathrop and T. B. Munroe (U.S.P. 1,572,539, 9.2.26. Appl., 20.6.24).—Bagasse and similar fibres are softened by suitable fermentation (cf. following abstract), and so piled that the heat generated partially dries and sterilises the material.

A. Geake.

Preserving fibres for pulp making. E. C. Lathrop and T. B. Munroe (U.S.P. 1,572,540, 9.2.26. Appl., 20.6.24).—By wetting green bagasse and similar fibres the growth of moulds and fungi is inhibited, and that of lactic acid-producing and similar bacteria is promoted. This fermentation is continued until the desired proportion of parenchymatous matter is destroyed. A. Geake.

Production of pulp. L. Bradley and E. P. McKeefe (U.S.P. 1,572,840, 9.2.26. Appl., 10.4.24). —Chemical pulp is boiled with sodium hydroxide and sodium thiosulphate, the former being considerably in excess of that required to decompose silicates and aluminates in the material treated. A. Geake.

Cellulose from vegetable substances. J. M. Schmidt (Can. P. 247,077, 29.12.23).—The fibres of peat or the like are loosened by heating or freezing, freed completely or partly from colloidal constituents, and treated many times alternately with alkali and chlorinating baths with vigorous stirring.

A. Geake.

Paper pulp from seaweed. A. J. and H. de Montby (F.P. 595,394, 17.6.24).—The seaweed is washed with sulphuric acid, hydrochloric acid, and soft water. It is then treated in a rotating washer or autoclave with a solution of sodium hydroxide and quillaia bark and steam at 3—4 atm. pressure for 8—10 hrs. After pressing and washing with soft water, it is treated with chlorine and acidified.

A. Geake.

Replacing a heavy or cold liquid containing cellulose, peat, or the like, in suspension, by a light or hot liquid. T. N. M. Molin (G.P. 416,600, 1.8.23. Conv., 9.8.22).—The mass, *e.g.*, pulp from a cellulose boiler, is run into a diffusion vessel with a false bottom, and the light or hot liquid introduced at the top. The heavy or cold liquid runs away through the false bottom, and is replaced by the light or hot liquid. The mass may then be more readily pressed. A. Geake.

Utilising the condensate from indirectly-heated sulphite-cellulose boilers. R. KARLBERG (G.P. 420,830, 19.11.24. Conv., 27.11.23).—The condensate is treated with manganese dioxide to oxidise and neutralise the acids ; neutralisation is completed with sodium hydroxide or carbonate. Heat losses are avoided by carrying out the operations in a closed vessel under pressure. A. GEAKE.

Reclaiming used paper. G. HAMMOND (U.S.P. 1,572,478, 9.2.26. Appl., 12.3.25).—Paper, to which a pigment has been applied, is recovered by heating with caustic alkali below 43°, and then adding an alkali hydrogen sulphite at the ordinary temperature. A. GEAKE.

De-inking paper. G. HAMMOND (U.S.P. 1,572,479, 9.2.26. Appl., 24.5.22).—A composition for de-inking paper comprises a saponaceous ingredient, an ingredient of the glucose series, and water. A. GEAKE.

Recovering waste sulphuric acid from manufacture of parchment paper. H. BECHHOLD and H. KARPLUS (G.P. 418,831, 10.7.24).—The sulphuric acid is concentrated until suitable for the parchmentising process and treated with a solid adsorbent, such as silica or a silicate. Alternatively concentration is omitted, and the acid only heated before treatment with an absorbent. A. GEAKE.

Manufacturing threads of artificial silk of highest degree of fineness from viscose. L. HESSE and H. RATHERT (U.S.P. 1,575,052, 2.3.26. Appl., 13.2.22).—See E.P. 240,717 ; B., 1925, 986.

Pumps [for use in manufacture of viscose silk]. E. LUNGE, and COURTAULDS, LTD. (E.P. 248,038–41, 20.11.24).

Apparatus for manufacture of artificial silk. E. LUNGE, and COURTAULDS, LTD. (E.P. 248,042 and 248,046, 20.11.24).

Air-vessels for securing regular flow from reciprocating pumps [in manufacture of viscose silk]. E. LUNGE, and COURTAULDS, LTD. (E.P. 248,045, 20.11.24).

Washing textile material on bobbins (E.P. 242,612).—See VI.

Manufacture of alkali carbonates (Can.P. 248,096).—See VII.

Sugar from cellulose (F.P. 595,439).—See XVII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Theory of the dyeing process. E. ELÖD (Melliands Textilber., 1925, 6, 742—743 ; Chem. Zentr., 1926, I, 1039—1040).—The equilibrium hydrogen-ion concentration of solutions of acid or basic dyes in contact with wool is characteristic of the wool used and depends neither on the concentration of the dye nor on the initial p_H of the solution. The quantity of dye taken up is not directly connected with the change in p_H over the region investigated. No marked decomposition of wool occurs with solutions having p_H equal to the so-called isoelectric point of the wool, whereas the wool proteins are broken down at hydrogen-ion concentrations removed from this value. The isoelectric point varies from one kind of wool to another and, since it is a resultant of the isoelectric points of the amphoteric substances composing the wool, may not be well defined even for an individual sample. L. M. CLARK.

Physics and chemistry of the dyeing process. K. H. MEYER (Melliands Textilber., 1925, 6, 737—739 ; Chem. Zentr., 1926, I, 1040).—The theory that dyeing may be due to solution of the dye in the fabric, is supported by the facts that the amount of *o*-nitroaniline taken up by cellulose acetate silk from aqueous solution depends on the concentration, and that the colour of a fibre so treated is uniform throughout the cross-section of the fibre. Moreover, all substances, coloured or not, which can be extracted from aqueous solution by ethyl acetate, penetrate acetate silk. With basic dyes, the term "adsorption" is justified, since here the dyes are attached only to the surface of the material ; the aromatic nuclei of the dyes may be supposed to penetrate the fibre, while the ammonium radical is turned towards the aqueous medium. When the mordant, "Beize für Acetatseide," B.A.S.F., is used, both solution (as mordant–dye compound) and adsorption of the dye may take place. L. M. CLARK.

Physical changes of dyes within dyed fibres. R. HALLER and A. RUPERTI (Cellulosechem., 1925, 6, 189—192).—Cellulose acetate silk dyed at low temperatures with Para-Red has a yellowish shade, the dye being uniformly distributed within each fibre ; after immersion in hot or boiling water, the shade becomes redder and the dye agglomerates into small particles. Similar results are obtained, although with greater difficulty (steaming under pressure is necessary), when the Para-Red is obtained from Naphthol AS (the anilide of β-hydroxynaphthoic acid) instead of β-naphthol or when aminoazobenzene is used instead of *p*-nitroaniline. Similar changes are observed in Chardonnet silk dyed with the same dyes and also with indigo, Thioindigo Red, and Indanthrene Blue. Chardonnet silk dyed cold with Naphthylamine Claret (α-naphthylamine diazotised and coupled on the fibre with β-naphthol) contains the dye evenly distributed ; when heated in water under 1 atm. pressure the dye agglomerates slightly without change of shade, but when heated for a prolonged period in boiling water or subjected to a short steaming under 6 atm. pressure, agglomeration becomes complete and the dye migrates towards the surface of each fibre and is there deposited as well-defined crystals which may be removed by washing and pressing, the fibres being thereby decolorised. Thioindigo Red dyed on Chardonnet silk behaves similarly. Chrome Yellow (from lead acetate and a dichromate) dyed on Chardonnet silk is at first evenly distributed but after steaming under 4 atm. pressure it agglomerates and becomes orange (even in the absence of an alkali) although no migration of the pigment occurs. Similar changes are observed by

steaming dyed cotton, except that the agglomerated dyes migrate to the boundaries of the lumen in each fibre as well as to the cuticle, the migration, change of shade, and condensation or crystallisation of the particles of dye being favoured by prolongation of the steaming or rise of temperature. Vat dyes, Indigo and Thioindigo Red easily, and Indanthrene Red 5 GK, Indanthrene Brilliant Violet RK, and Indanthrene Blue RS with greater and increasing difficulty, crystallise and migrate within cotton fibres to the lumen and cuticle when steamed, accompanied by a change in shade. Uncertain results are obtained by steaming cotton dyed with direct dyes. Alizarin Red dyed on cotton mordanted with aluminium acetate is evenly distributed within each fibre, but when steamed for 1 hr. under 0·5 atm. pressure the pigment agglomerates and migrates to the lumen and cuticle; that deposited near the cuticle is removed by washing with water, the fibre being left colourless; under similar conditions the presence of Turkey-red oil considerably retards the agglomeration and migration, and the dye which migrates to the cuticle cannot be removed by washing. The decrease in fastness to rubbing produced by steaming cotton dyed with indigo is due to migration of the dye to the cuticle of each fibre. A. J. HALL.

PATENTS.

Weighting silk. DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H. (G.P. 422,097, 17.10.17. Addn. to 320,783).—Zirconium salts are used instead of tin salts for weighting silk by the process described in the chief patent (cf. E.P. 116,102; B., 1920, 17 A). For example, silk is treated with zirconium nitrate solution, *d* 1·21, followed, after centrifuging, by treatment with ammonia, which may be diluted with inert gases. L. A. COLES.

Washing, bleaching or dyeing textile materials wound on perforated bobbins. NAAML. VENNOOTS. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 242,612, 19.10.25. Conv., 8.11.24).—The bobbins, immersed in the dye-liquor or other liquid, are attached to a long vertical pipe, the column of liquid in the pipe supplying the suction necessary to draw the liquid through the material. The process is suitable for bleaching, dyeing, etc., and for washing out acid from freshly spun viscose silk. L. A. COLES.

Apparatus for dyeing and otherwise treating textiles, textile fabrics, and the like. P. STAPLETON and E. STROUD (E.P. 246,386, 24.7.25).—A dye vat with a curved or sloping bottom is provided with a steam injector by means of which dye liquor is withdrawn from the upper part of the vat, through a small reservoir, and is subsequently discharged through perforations in the upper side of a horizontal pipe located at the lowest part of the vat, thereby promoting uniform circulation of liquor and textile materials within the vat. The injector is provided with a by-pass which allows the dye liquor to be heated independently of its circulation. A. J. HALL.

Processes in which cellulose acetate artificial silk and like products are treated with hot liquors. SILVER SPRINGS BLEACHING AND DYEING CO., LTD., and A. J. HALL. (E.P. 246,879, 31.7.24).—Cellulose acetate silk may be treated for prolonged periods in boiling aqueous liquors without becoming curly and wool-like and without loss of lustre and transparency if the liquor contains not less than a certain minimum amount of a protective salt, the minimum quantity being dependent on the particular salt or salts used. Suitable salts include sodium, ammonium, calcium, magnesium, barium, aluminium, strontium, and potassium chlorides; ammonium, sodium, copper, magnesium, zinc, and potassium sulphates; sodium sulphite, alum, chrome alum, sodium chlorate, potassium oxalate, and sodium nitrate. The dyeing properties of the cellulose acetate silk are not altered by such treatment. In general, 10—30% solutions of the protective salts are used and they are particularly useful in the treatment of cellulose acetate materials containing other fibres in the dyeing, bleaching, scouring, finishing, or other treatment in which it is necessary to use boiling liquors or liquors at a temperature exceeding 85°. The process has particular application in the dyeing of cellulose acetate-wool union textile materials. A. J. HALL.

Dyeing acetyl cellulose [cellulose acetate] or fabrics containing it and new products [dispersing agents] for use therein. BRIT. DYESTUFFS CORP. LTD., J. BADDILEY, A. SHEPHERDSON, H. SWANN, J. HILL, and L. G. LAWRIE (E.P. 246,984, 7.1.25. Addn. to 224,077; B., 1925, 39).—Much smaller quantities (*e.g.*, less than 10% calculated on the weight of dry dye) of the dispersing agents consisting of condensation products of naphthalene with formaldehyde as described in the chief patent are utilised for the preparation of solubilised dyes for cellulose acetate silk. For example, a dyestuff paste consists of 10 pts. of aminoanthraquinone, $\frac{1}{4}$ pt. of the dispersing agent, and $89\frac{3}{4}$ pts. of water. Further, complete neutralisation of acidic dispersing agent in the solubilised dyestuff paste by addition of ammonia is preferred to the partial neutralisation with caustic soda described in the chief patent. A. J. HALL.

Dyeing cellulose esters. BADISCHE ANILIN- & SODA-FABR., Assees. of F. GÜNTHER and F. LANGE (G.P. 420,017, 12.6.23).—Dyeing is carried out with the water-soluble sulphamic acids of the dye, and the colours obtained may be diazotised and developed with chromogens or coupled with diazo-compounds. Thus, the azo dye from *p*-aminophenylsulphamic acid and β-naphthol, used in acid solution, gives brownish-red shades, while further diazotisation and treatment with β-naphthol produces a red-violet colour. L. M. CLARK.

Dyeing of textile fabrics [with basic dyes]. W. EBERLEIN, and COLLOISIL COLOUR CO., LTD. (E.P. 247,328, 29.11.24).—Textile materials are treated with colloidal suspensions of natural or artificial silicates insoluble in water, such as "green earth" or "fixing earth," before they are dyed with basic dyes or printed with pastes containing

the corresponding colour bases. Alternatively, the dye and colloidal material may be contained in the same bath. The colour lakes precipitated on the fibre are subsequently fixed, *e.g.*, by heating the material or by treating it with an electrolyte. Protective colloids, such as glue, casein, or albumin, may be added to the colloidal suspensions, in which case the material is subsequently treated with dilute formaldehyde, which may be added to the dye-bath. The dyeings produced are fast to light.
L. A. COLES.

Bleaching and dyeing furs and the like. H. STEIN, W. E. AUSTIN, and I. LIEBOWITZ, Assrs. to STEIN FUR DYEING CO. (U.S.P. 1,573,200, 16.2.26. Appl., 3.5.24).—Animal fibres, particularly fur skins, are bleached with hydrogen peroxide solution in the presence of a fibre-protecting agent comprising ferrous sulphate, before they are dyed.
L. A. COLES.

Azo dyes stable in steam from nitrosamine colours. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of J. GÜRTLER (G.P. 419,431, 20.5.24).—Addition to the printing colours of aromatic nitro- or nitroso-compounds as oxidising agents, permits the development of the dye in full strength and purity of tone, even where prolonged steaming is required. Suitable compounds include nitrosulphonic acid salts of the benzene, naphthalene, and anthraquinone series as well as their halogen-substituted derivatives, and salts of nitrophenolsulphonic and nitroso-naphtholsulphonic acids. L. M. CLARK.

Developing salts. BADISCHE ANILIN- & SODA-FABR., Assees of H. KRZIKALLA (G.P. 421,837, 16.8.24).—Claim is made for anhydrous mixtures of salts of mineral acids, diazotisable aromatic amines (neither nitrated nor sulphonated), and nitrites, to which, if necessary, other acid or indifferent substances are added. The diazo solutions produced on dissolving the mixtures in cold water, can be used immediately for the production of dyes on the fibre. A. COULTHARD.

Production of effect threads from animal fibres. FARBENFABR. VORM. F. BAYER & CO., Assees. of G. RUDOLPH (G.P. 422,464, 26.2.24. Addn. to 407,834).—Very pure effects are obtained by using formaldehyde instead of formic acid, and reducing the proportion of tin salts, in the process described in the chief patent (cf. B., 1925, 240).
L. A. COLES.

Dyeing leathers tanned differently from glacé leather. L. CASSELLA & CO., Assees. of F. KOHL (G.P. 422,465, 6.12.23).—The leather is either treated with tanning materials neutralised with alkali immediately before dyeing, or it is dyed from a bath containing both the neutralised tanning material and the dye. Under these conditions, the original tannage is very little affected and level dyeing results. A. COULTHARD.

Improving the fastness to light of insoluble dyes produced on the fibre from arylides of 2:3-hydroxynaphthoic acid which contain a hydroxyalkyl group in the molecule. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of B. SCHÄFER (G.P. 422,468, 3.7.24).—The dyeings are treated with solutions of metal salts above 100° under pressure. For example, dyed cotton is heated for ½ hr. under about 1 atm. pressure with solutions containing 2—3 g. of sulphate of copper, zinc, or nickel, per litre. A. COULTHARD.

Process of dyeing. W. DUISBERG and W. HENTRICH, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,575,324, 2.3.26. Appl., 26.11.24).—See E.P. 225,862; B., 1925, 583.

Dyeing apparatus. B. K. THIES (U.S.P. 1,576,378, 9.3.26. Appl., 23.10.24).—See E.P. 232,494; B., 1925, 497.

Dyeing and printing of textile goods and other materials. DURAND ET HUGUENIN S.A., Assees. of M. BADER, T. LOMBARD, C. SUNDER, and C. VAUCHER (U.S.P. 1,575,958, 9.3.26. Appl., 24.7.23).—See E.P. 203,681; B., 1923, 978 A.

Artificial silk (E.P. 245,815).—See V.

Waste waters from dyeworks (Austr. P. 100,735).—See XXIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Caro's reagent. R. H. VALLANCE (J.S.C.I., 1926, 45, 66 T).—Solutions of potassium permonosulphate, approximating to gram-molecular concentration, have been prepared by triturating potassium perdisulphate with concentrated sulphuric acid below 0°, and neutralising with potassium carbonate under specified conditions.

Catalytic oxidation of ammonia. L. ANDRUSSOV (Z. angew Chem., 1926, 39, 321—332). —The composition of the issuing gases, after passing a mixture of air and ammonia containing 8·3—9·0% NH_3 over a platinum gauze catalyst, has been determined for various temperatures and many different rates of flow. Combustion of the ammonia commences at 160°, at which temperature nitrogen is the chief product, although small quantities (3—6%) of nitric oxide are always present. At and above 400° practically the whole of the ammonia is decomposed. The amount of nitric oxide formed increases with rise of temperature until at 550° this gas is the chief product of the reaction. At 500° side-reactions involving double decomposition of ammonia with nitric oxide as well as thermal decomposition of part of the ammonia and part of the nitric oxide take place, and the proportion of the reaction products consumed in these reactions increases rapidly with rise of temperature and inversely with the velocity of the gases over the catalyst; with a sufficiently high velocity of the gas stream, however, a 90% yield of nitric oxide may be obtained, even at 1000°. These results may be satisfactorily explained on the assumption that

an intermediate product, NOH, is formed which may decompose into nitrogen, water, and hydrogen, into nitric oxide and water, or, thermally, into nitrogen and water. A. R. POWELL.

Determination of ammonia in ammoniacal and industrial liquors. C. JUNGBLUT (Bull. Soc. chim., 1926, 39, 336—337).—The following method for the determination of ammonia in ammoniacal liquor avoids the brown discoloration produced when carbon dioxide is expelled by prolonged boiling after acidification with sulphuric acid. The liquor containing the ammonia chiefly in the form of bicarbonate is treated with sodium hydroxide and then with barium chloride. The barium carbonate is rapidly filtered off, the filtrate made neutral to methyl-orange, and the ammonia determined in the usual way by treatment with formaldehyde to convert the ammonia into hexamethylenetetramine, followed by titration of the liberated acid, using phenolphthalein as indicator.

W. HUME-ROTHERY.

Analytical control in permanganate manufacture. H. WALDE and K. A. SCHUCH (Wiss. Veröff. Siemens-Konz., 1925, 4, 188—199).—The method of Aschoff (cf. Sackur, Ber., 1910, 43, 381, 448) is found to be the most satisfactory for determining potassium manganate volumetrically, the alkaline solution being added at 70—90° to the standard oxalic acid solution containing an excess of sulphuric acid. The end-point is made sharper by the addition of phosphoric acid. The titration was examined electrometrically and the critical potential found to be 0·796 volt measured against a normal calomel electrode. The use of a comparison electrode with a capillary electrometer as zero instrument is also described. The potentiometric and visual methods of determining the end-point were compared in the titration of solutions at various dilutions, the results being in good agreement, but the potentiometric method is to be preferred for dilute solutions. To determine the free alkali in the manganate solutions, the manganate and permanganate are destroyed by addition of hydrogen peroxide, the solution filtered from the precipitated manganese dioxide, and titrated with standard acid. The alkali produced in the decomposition of the manganate must be subtracted from the value observed, and a further correction is necessary for the potassium hydroxide adsorbed by the hydrated manganese dioxide precipitate. The adsorption was found to be 1·039 g. of potassium hydroxide per g. of manganese in 5*N*-alkali, falling to 0·905 g. per g. of manganese in 0·5*N*-solution. For the determination of permanganate in presence of manganate a method is suggested based on the quantitative decomposition of the manganate by carbon dioxide: $3K_2MnO_4+2CO_2\rightarrow 2KMnO_4+MnO_2+2K_2CO_3$. The manganese dioxide is separated, and determined by conversion into pyrophosphate, and the total permanganate is determined by titration. The original permanganate is then obtained by difference, by deducting the quantity of permanganate corresponding to the manganese dioxide. G. M. BENNETT.

Commercial calcium carbide. I. E. SCHLUMBERGER (Z. angew. Chem., 1926, 39, 213—220).—Microscopical examination of polished surfaces of samples of commercial calcium carbide of varying composition shows that the calcium carbide–calcium oxide eutectic lies in the neighbourhood of 60% of carbide. Calcium oxide crystals cannot, however, be recognised until the carbide content is reduced to about 40%. Products containing 60—40% of carbide show small calcium carbide crystals surrounded by a eutectic mass. The relation between the density and carbide content is a linear one. Measurements of the electrical resistance of commercial calcium carbide show that this varies according to the direction of the current relatively to the arrangement of the carbide crystals as seen microscopically. With decreasing carbide content the resistance increases and reaches a maximum at 65—70% of carbide. Below this, however, it rapidly decreases again, and a 40% carbide conducts electricity 400 times better than a 94% carbide. This may be due to the presence of impurities, but not of graphite (though this is present), since the temperature coefficient of commercial carbide between 25° and 125° is greater than that of graphite. It remains to be proved, indeed, whether absolutely pure calcium carbide is really a conductor of electricity. The fact that commercial carbide of low carbide content reacts more readily with nitrogen is ascribed to the smaller size of the carbide crystals in it, as seen from photomicrographs, with which the work is illustrated. W. T. K. BRAUNHOLTZ.

Analysis of silicates. R. SCHWARZ and A. SCHINZINGER (Z. anorg. Chem., 1926, 151, 214—220).—The silicate is decomposed by fusion with lithium carbonate in a platinum crucible. The product is treated as in the sodium carbonate method except that traces of platinum dissolved from the crucible must be removed with hydrogen sulphide before precipitating aluminium, and magnesium is determined volumetrically by the method of Klingenfuss (B., 1924, 852). Small quantities of sodium and potassium are separated from the lithium salts by first precipitating the bulk of the latter as phosphate, then applying the Gooch method. R. CUTHILL.

Helium for divers. G. GAERTNER (Umschau, 1926, 30, 50—21 ; Chem. Zentr., 1926, I., 1871—1872).—Nitrogen at high pressure is the cause of diving sickness, whilst pure oxygen at pressures above 2·5 atm. acts on the lungs as a rapid poison. Helium is innocuous and suitable for the dilution of the oxygen. C. IRWIN.

PATENTS.

Manufacture of sulphuric acid. METALLBANK U. METALLURGISCHE GES., Assees. of H. KLENCKE (G.P. 421,786, 3.6.24).—Deposition of sulphuric acid from mixtures of gases containing sulphur dioxide with a maximum proportion of oxides of nitrogen, is effected in such a manner that solution or precipitation of the oxides of nitrogen is prevented, *e.g.*, by the influence of an electrostatic field. L. A. COLES.

Manufacture of sulphuric acid. RHENANIA VEREIN CHEM. FABR. A.-G., ZWEIGNIEDERLASSUNG MANNHEIM, Assees. of W. FRANK (G.P. 422,572, 6.3.23. Addn. to 406,490).—The Gay-Lussac and Glover towers used in the process described in the chief patent (*cf.* E.P. 212,768; B., 1924, 419), contain no filling material. L. A. COLES.

Manufacture of sulphur dioxide and sulphuric acid from sulphur-bearing slag. GEWERKSCHAFT LUTZ III (G.P. 423,640, 23.1.24).—Enriched air or oxygen is blown through the slag. The gases, with their excess oxygen, after treatment for recovery of acid, are used in the blast furnace. C. IRWIN.

Removing arsenic from burner gases. METALLBANK U. METALLURGISCHE GES. A.-G., Assees. of O. KURZ (G.P. 423,657, 21.3.24. Addn. to 368,283; B., 1923, 400 A).—After the electrical precipitation of flue-dust the gas is cooled sufficiently to form clouds but without addition of moisture. Arsenious oxide is then deposited electrically as dust, prior to the treatment in presence of sulphuric acid described in the chief patent. C. IRWIN.

Production of phosphoric acid and hydrogen. F. G. LILJENROTH (F.P. 595,987, 31.3.25. Conv., 15.4.24).—Metal phosphides reducible by hydrogen are treated at raised temperatures with water or steam, oxygen being added if necessary. L. A. COLES.

Manufacture of phosphoric acid. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of R. SUCHY (G.P. 423,275, 18.1.25).—Phosphorus or gases containing phosphorus are burnt in a closed vessel, the walls of which are continuously moistened with phosphoric acid solution. A. R. POWELL.

Manufacture of arsenic acid and arsenates. BADISCHE ANILIN- & SODA-FABR., Assees. of J. BRODE and K. KLEIN (G.P. 423,276, 1.1.25). Arsenic trioxide or an arsenite is heated under pressure with water and gases containing oxygen to a temperature above the b.p. of the solution. A. R. POWELL.

Utilisation of the energy liberated during the oxidation of hydrogen compounds of, *e.g.*, sulphur, nitrogen, and phosphorus, to mineral acids. E. R. BESEMFELDER (G.P. 421,665, 9.3.20).—Mixtures of oxygen with hydrogen compounds of, *e.g.*, sulphur, nitrogen, or phosphorus, are exploded in the cylinder of a gas engine, and the exhaust gases are cooled and condensed in suitable heat-exchanging apparatus. The explosive power of the mixtures may be modified by the addition of non-reacting gases. L. A. COLES.

Manufacture of hydrochloric acid and magnesia from magnesium chloride. E. FRANK (G.P. 422,322, 24.8.24).—Magnesium chloride which has been partially dehydrated by heating to 500—600° in a rotating furnace, is partly decomposed by external heating alone and decomposition is completed by external heating aided by treatment with a counter-current of steam. L. A. COLES.

Shaft furnaces [for burning limestone etc.]. E. CORNET (E.P. 245,653, 16.8.24).—The object of the invention is to ensure the combustion of carbon monoxide which is always generated in shaft furnaces for burning limestone etc. Air, preheated by waste heat in flues disposed in the furnace wall, passes thence into vertical flues in the refractory lining of the furnace. These flues are controlled by dampers and have outlets leading directly into the furnace in the zone most favourable to combustion of carbon monoxide and also in the calcining zone. Preheated air may also be introduced centrally into the furnace. R. B. CLARKE.

Lime-kiln. H. MISCAMPBELL (U.S.P. 1,572,156, 9.2.26. Appl., 16.2.25).—A lime-kiln consists of a vertical shaft with two parallel walls and two downwardly diverging walls, a port for injecting heating gas being arranged in the lower portion of the shaft. A. R. POWELL.

Treatment of aluminous materials. H. SPENCE, W. B. LLEWELLYN, and P. SPENCE AND SONS, LTD. (E.P. 247,078, 6.8 and 18.9.24).—Aluminium sulphate is prepared by circulating sulphuric acid through aluminous material, such as shale, silicious bauxite, and the like, in small lumps provided these are fairly hard and contain sufficient silica to prevent disintegration. Friable bauxite and the like cannot be used directly as the circulation would be blocked. In order to make such materials available they are agglomerated with plastic clay, sodium silicate, or other suitable binder and baked at a dull red heat. The pieces so produced may be broken so as to pass a ½-in. sieve and then extracted as usual. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1917 and 1919, to E.P. 119,924 and 112,881.) C. IRWIN.

Production of alkali silicates soluble in water. B. E. D. KILBURN. From NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (E.P. 247,439, 16.5.25).—Hydrated silicic acid prepared by treating leucite or a similar silicate with nitric acid is heated with an alkali nitrate or nitrite. Reaction commences at 250° and is complete at 600—700° without melting. The nitrous gases evolved are recovered and the porous solid product is dissolved in water and the solution filtered. Diatomaceous earth may also be used as a source of silica. C. IRWIN.

Alkali silicate powder. F. FINZI (F.P. 597,825, 5.3.25).—Solutions of alkali silicates are atomised by the aid of compressed air and a current of hot air. A. R. POWELL.

Treating [purifying] zinc sulphate solutions. R. B. ELDRIDGE (U.S.P. 1,573,233, 16.2.26. Appl., 13.10.24).—Inorganic impurities are precipitated by the action of a soluble hyposulphite, and the solution is filtered. C. IRWIN.

Producing borax and sodium bicarbonate from lake brines. M. V. LOWRY, Assr. to WEST END CHEMICAL CO. (U.S.P. 1,573,259, 16.2.26. Appl., 10.4.23).—The brine is concentrated by evaporation, carbon dioxide passed in, and the mixture cooled. Borax and sodium bicarbonate

separate as a sludge. This is mixed with colemanite and heated to boiling point. C. Irwin.

Producing light basic magnesium carbonate. R. B. Crowell, Assr. to Western Industries Co. (U.S.P. 1,573,632, 16.2.26. Appl., 26.6.25).—Magnesium oxide in aqueous suspension is treated with carbon dioxide until the ratio of CO_2 to MgO lies between 0·8 and 0·9. The product is then heated to a temperature not above 60°. C. Irwin.

Manufacture of alkali carbonates. L. Bradley and E. P. McKeefe (Can.P. 248,096, 18.6.23).—Material containing cellulose is digested with neutral alkali sulphite solution, the solution, after separation of the cellulosic material is evaporated to dryness, and the residue is calcined and fused. L. A. Coles.

Treatment of natural alkali salts of secondary and tertiary origin. A. Lambert (Can.P. 249,255, 12.6.24).—A solution of sodium chloride, sulphate, and carbonate obtained from native deposits is treated with carbon dioxide to precipitate sodium hydrogen carbonate, the mixture is heated to 330° under pressure, and the salt separated from the liquid without cooling. A. R. Powell.

Decomposition of sodium ammonium sulphate. E. A. E. Waché (F.P. 594,694, 31.5.24).—Sodium ammonium sulphate and sodium chloride are added in the proportions required by the equation, $Na_2(NH_4)_2(SO_4)_2,4H_2O+2NaCl=2Na_2SO_4+2NH_4Cl+4H_2O$, to a solution saturated at 30° with ammonium chloride and sodium sulphate, in such quantities that anhydrous sodium sulphate but no ammonium chloride is thrown out of solution on heating to 90—100°. After removing the sodium sulphate, the solution is cooled, ammonium chloride which crystallises out is removed, and the mother liquor is used again in the process. L. A. Coles.

Ammonium sulphate saturator. Soc. de Fours à Coke et d'Entreprises Ind. (F.P. 596,494, 9.4.25).—A saturator for the manufacture of ammonium sulphate is provided with a centrifugal pump in the axis of the saturator above the floor. The pump is driven by a motor on the roof of the saturator and serves to prevent settling of the ammonium sulphate on the bottom. A. R. Powell.

Bleaching and purifying alkaline-earth sulphates, especially barium sulphate. A. A. Ackermann (F.P. 596,640, 15.4.25).—Alkaline-earth sulphates are purified by treatment with hydrogen fluoride or compounds derived from it. L. A. Coles.

Preparation of the gas mixture for ammonia synthesis. E. Edwin (F.P. 596,714, 11.4.25).—A mixture of steam and the vapour of a hydrocarbon is subjected to the action of the electric arc and the resulting mixture of carbon monoxide and hydrogen is passed together with steam and air into a gas-producer. Part of the issuing gases is passed back over the arc, while the remainder is subjected to the action of a catalyst to convert the carbon monoxide and steam into carbon dioxide and hydrogen, the former being subsequently removed by known methods. A. R. Powell.

Recovery of vanadium compounds from iron ores containing vanadium and titanium. B. P. F. Kjellberg (F.P. 598,315, 16.5.25. Conv., 9.11.24 and 12.2.25).—The ore is roasted in such a way that the vanadium compounds are prevented from oxidising to vanadium pentoxide and the roasted material is then leached with a solvent for the vanadium. A. R. Powell.

Preparation of pure bismuth vanadate. E. Zintl and L. Vanino (G.P. 422,947, 9.8.24).—Bismuth vanadate for therapeutic use is precipitated by the addition of vanadates, at the ordinary or at raised temperatures, to solutions containing bismuth salts and substances which prevent the separation of basic bismuth compounds, such as acetic acid, mixtures of acetic acid and acetates, small quantities of mineral acids, polyhydric alcohols, or ketones. The product is subsequently heated in the air in case any reduction has taken place during the precipitation. L. A. Coles.

Adsorption of sulphur dioxide from gas mixtures. M. Schroeder (G.P. 421,725, 13.8.24).—Gas mixtures containing sulphur dioxide are treated under pressure with a counter-current of water for absorbing the sulphur dioxide, in a tower in which by regulating the escape of the unabsorbed gases, the pressure is maintained constant throughout the process. The expansibility of the residual gas, after passage through heat-exchangers in which it is heated by the heat of compression of the freshly compressed gas or by other means, is utilised, *e.g.*, in expansion cylinders, for compressing fresh supplies of the gas before entry into the tower. The pressure prevailing in the absorption tower forces the sulphur dioxide solution continuously into apparatus at the top of the tower, where the sulphur dioxide is expelled by heating. L. A. Coles.

Production of potassium nitrate. Wolff u. Co., Assees. of H. Hampel (G.P. 421,988, 24.5.21).—Potassium nitrate is produced by interaction of potassium chloride and calcium or magnesium nitrate in the cold. L. A. Coles.

Production of finely divided iron oxide. Badische Anilin- & Soda-Fabr., Assees. of A. Mittasch (G.P. 422,269, 24.5.24).—The product is obtained by burning iron carbonyl, or mixtures of it with other combustible gases, or with inert gases. L. A. Coles.

Preparation of insoluble silver alkali thiosulphates. Chem. Fabr. Schleich G.m.b.H., and A. Rosenheim (G.P. 422,323,7.8.24).—Insoluble silver alkali thiosulphates are produced by adding approximately the calculated quantity of silver salts, particularly silver halides, to concentrated alkali thiosulphate solutions at 30°, the mixture being thoroughly stirred. The insoluble compounds are rendered water-soluble by agitation with highly concentrated alkali thiosulphate solution at 0°. L. A. Coles.

Production of alkaline-earth chlorides. Verein für Chem. u. Metall. Produktion (G.P. 422,470, 17.7.23).—Alkaline-earth chlorides are produced by heating alkaline-earth carbonates with solid magnesium chloride. L. A. Coles.

Production of barium, strontium, and calcium peroxides. H. Schulze (G.P. 422,531, 19.2.24).—Hot or cold solutions containing alkaline-earth sulphides or mixtures of these with alkaline-earth hydroxides, are electrolysed in an open or closed anode chamber, which is separated from the cathode chamber, using anodes constructed of iron or other metals, or of carbon. Air free from carbon dioxide, ozonised air, oxygen, or "active oxygen" may be passed continuously or intermittently into the solution. L. A. Coles.

Evaporation of brine. Maschinenbau-A.-G. Balcke (G.P. 422,657, 25.4.22).—Brine is concentrated until gypsum settles out, in pans heated directly by furnace gases, and is subsequently concentrated to deposit salt, in evaporators heated by steam generated during the first stage of the evaporation; the steam is first superheated by waste combustion gases from the furnace. The heat of the furnace gases is also used for preheating the brine charged into the pans. L. A. Coles.

Preparation of brine free from gypsum. Salzbergwerk Neustassfurt (G.P. 423,861, 26.11.24).—To the water used for dissolving impure rock salt containing gypsum such salts are added as diminish the solubility of calcium sulphate. These salts may be added to the brine after evaporation in the pans or used in the boreholes etc. C. Irwin.

Apparatus for the manufacture of hypochlorite solutions. Badische Anilin- & Soda-Fabr. (G.P. 422,725, 3.3.25).—A vessel with a non-metallic surface is surrounded by an outer jacket containing a non-reacting liquid in which is immersed a tube supplied with liquid chlorine, and the chlorine gas generated by its evaporation is conveyed through non-metallic tubes into alkali solution contained in the inner vessel. L. A. Coles.

Production of ammonia, sulphur, and thiosulphates. Fabr. Chem. Prod. F. Hefti, and W. Schilt (G.P. 422,726, 23.10.23. Conv., 24.9.23).—The products are obtained by reducing nitrites, especially alkali nitrites, with two mols. of hydrogen sulphide. L. A. Coles.

Complete utilisation [of the end liquors from] crude potassium salts. Wolff & Co., and F. Frowein (G.P. 422,987, 24.8.24).—The liquors are subjected to two cycles of treatment in one of which the sulphate ions are converted into ammonium sulphate with the aid of calcium carbonate and in the other of which the chlorine ions are converted into ammonium chloride with the aid of lead carbonate. Sodium, potassium, and magnesium nitrates are recovered during the course of the process. The magnesium nitrate is converted into a fine-grained, stable fertiliser by mixing the concentrated mother liquors with potassium sulphate at 60—70°, and stirring, kneading, and finally grinding the mixture. A. R. Powell.

Manufacture of alkaline-earth nitrides. F. Uhde (G.P. 423,348, 6.12.21).—The oxides, hydroxides, or carbonates of alkaline-earth metals are converted into nitrides by treating them with hydrogen or hydrocarbons in an electric furnace and subjecting the metallic regulus so obtained to the action of nitrogen. A. R. Powell.

Working up barium sulphide. Rhenania Verein Chem. Fabr. A.-G. Zweigniederlassung Mannheim, and F. Rüsberg (G.P. 423,755, 8.9.22).—Crude barium sulphide is decomposed with hydrochloric acid or dilute nitric acid. The hydrogen sulphide evolved is absorbed in alkali sulphite or bisulphite in order to prepare alkali thiosulphate by the method of G.P. 370,593 (B., 1923, 499 a). C. Irwin.

Production of nitrogen peroxide from calcium nitrate. L'Azote Français, Soc. Anon. (Swiss P. 99,037, 7.10.21).—The gas is obtained by heating calcium nitrate above 500°, but below 650°, in the presence of oxygen. L. A. Coles.

Production of material containing silicon and carbon. Gebr. Siemens & Co. (G.P. 421,951, 11.12.21; F.P. 554,683, 2.8.22).—The material is obtained by heating, *e.g.*, for 5—10 hrs., mixtures of 3—5 pts. of silicon carbide, 4—8 pts. of sand, and 1—2 pts. of carbon, to 1700—2000°, preferably to 1900°. The properties, particularly the electrical conductivity, of the product are regulated by the duration of heating, the temperature, and the proportions of the constituents. The addition of sawdust to the constituents renders the product porous. L. A. Coles.

Production of corundum in electric furnaces. H. Vierheller (G.P. 422,105, 24.9.24).—In the production of corundum in the electric furnace, material evolving gases, containing, *e.g.*, peroxides, carbonates, or ammonium salts, preferably moulded into solid form together with iron filings, is immersed in the fused bath at the end of the fusion and reduction process. L. A. Coles.

Magnesium hydroxide. A./S. De Norske Saltverker (Nor. P. 39,564, 27.5.21).—Magnesium hydroxide is produced by the electrolysis of solutions of magnesium sulphate at temperatures above 90°, preferably at the b.p. A. R. Powell.

Recovery of neon and helium from the air. Ges. für Linde's Eismaschinen A.-G. (G.P. 417,572, 9.12.22).—The residual gases from the rectification of liquid air are further cooled under 10 atm. pressure to remove the bulk of the remaining nitrogen, and the uncondensed gas is passed, under pressure and at the same temperature, through a tube filled with adsorbent charcoal which removes the last of the nitrogen. The purified noble gases are then charged directly into steel cylinders. A. R. Powell.

Refining process for pearl essence. J. PAISSEAU (U.S.P. 1,576,454, 9.3.26. Appl., 22.12.23).—See E.P. 188,774; B., 1923, 55 A.

Manufacture of sodium formate from carbon monoxide. M. ENDERLI, Assr. to R. KOEPP & Co. (U.S.P. 1,574,875, 2.3.26. Appl., 28.2.23).—See E.P. 165,163; B., 1921, 583 A.

See also pages 303, **Separation of gaseous mixtures** (E.P. 232,986). 304, **Evaporation of solutions** (E.P. 247,346). 309, **Gas mixtures containing nitrogen** (Swiss P. 112,359). 331, **Kilns for lime burning etc.** (G.P. 416,143). 332, **Production of fatty acids, glycerin, and ammonium sulphate** (G.P. 421,438). 333, **Blanc fixe and sodium thiosulphate** (G.P. 420,251).

VIII.—GLASS; CERAMICS.

Physical properties of porcelain. K. WETZEL (Ber. Deuts. Keram. Ges., 1925, **6**, 23—40; Chem. Zentr., 1926, I., 762—763).—Addition to the normal porcelain mixture (50% of clay, 25% of quartz, 25% of felspar) of 3% of various ingredients gave the following results: Lime lowered the m.p. by 6 Seger cones, appreciable softening occurring at cone 10. This oxide produced a fairly low modulus of elasticity (6800 kg./sq. mm.), a resistance to bending shock of 2·06, a high value of coefficient of expansion, and a low resistance to temperature change. Much undissolved quartz could be detected microscopically, and the transparency at cone 10 was not very good. Magnesia gave the greatest depression of m.p. (to cone 17), the interval between softening and melting being approx. 280°. The modulus of elasticity was little changed (7590 kg./sq. mm.), whilst other properties were similar to those of the lime mixture. Addition of talc produced an interval between softening and melting of 420°, a modulus of elasticity of 8166 kg./sq. mm., and the highest resistance to bending shock (2·12). The coefficient of expansion and thermal endurance were about the same as with lime and magnesia. Dolomite gave similar results to magnesia but the transparency slowly increased with rise of temperature. Softening and melting results for fluorspar were similar to those for talc, but the modulus of elasticity was lower (6840 kg./sq. mm.). Calcium fluoride produced a resistance to bending shock of 2·07, a fairly high expansion coefficient, and a very small thermal endurance. Ferric oxide gave a low sintering temperature (1150°), but a large interval of approx. 470° between softening and melting. The modulus of elasticity was 7650 kg./sq. mm., the resistance to bending shock 1·87; the thermal expansion coefficient was high, the thermal endurance low, and transparency was poor. Zinc oxide raised the m.p. to cone 31, and gave a large interval between softening and melting (approx. 470°). The resistance to bending shock was 1·74, and the porcelain had the lowest thermal expansion coefficient (38×10^{-7}), this coinciding with high thermal endurance. Microscopical examination revealed no undissolved quartz. Mixtures containing alumina, titania, or zirconia sintered above 1200°. With titania and zirconia the m.p. was cone 27—28, with alumina cone 32. All three oxides, but particularly alumina increased the modulus of elasticity and the resistance to bending shock (TiO_2 1·75, Al_2O_3 1·86). The transparency with zirconia was very low, with titania nil. In all cases there was a large formation of mullite. A. COUSEN.

Pottery from the ancient "oppidum" of Vindalium (Vaucluse). L. DESVERGNES (Ann. Chim. Analyt., 1926, **8**, 40—41).—Two types of pottery found on the Sève mountain on the site of the ancient town of Vindalium, classed respectively as (1) soft and fine and (2) hard and coarse, had the following characteristics:—fracture (1) ash coloured and homogeneous, (2) yellow periphery and dark brown centre; *d* (1) 1·718, (2) 1·885; loss at red heat (1) 21·26, (2) 8·31; SiO_2 (1) 50·07, (2) 70·65; Al_2O_3 (2) 16·92, (2) 8·55; Fe_2O_3 (1) 6·46, (2) 9·49; CaO (1) 5·13, (2) 2·79; MgO (1) 0·13, (2) 0·21; Mn, traces in both samples. D. G. HEWER.

PATENTS.

Lead-free enamels. C. TOTOT-GIBARU (F.P. 597,146, 6.8.24).—An enamel flux free from lead consists of 2—20 pts. of silica, 5—35 pts. of zinc oxide, 30—70 pts. of crystallised borax, 2—20 pts. of alkali nitrate, 0—8 pts. of alkali fluoride, and 0—30 pts. of calcium carbonate. Tin oxide or antimony trioxide is used as opacifier. A. R. POWELL.

Manufacture of opacifiers for enamels. I. KREIDL (G.P. 422,600, 29.11.24. Conv., 24.11.24).—Zircon is decomposed by fusion with alkaline reagents and the product so obtained is treated with hydrofluoric acid to convert the combined and adsorbed silica into silicon fluoride. The amount of silica retained by the zirconia is a function of the temperature of fusion and of the quantity of alkali used in the fusion. A. R. POWELL.

Sifting of granular or pulverulent materials [potters' slip]. A. C. HARRISON (E.P. 248,121, 13.12.24).

Material containing silicon and carbon (G.P. 421,951).—See VII.

IX.—BUILDING MATERIALS.

Three-component system lime-sllica-alumina and hydraulic binding materials. R. GRÜN (Zentr. Bauverwalt., 1926, **46**, [1], Reprint. 3 pp.).—Ceramic bodies and cementitious materials consist essentially of lime silica, and alumina; the cementitious or hydraulic properties of the binding materials depend on their lime content, as represented by the calcium aluminates and calcium silicates present. The calcium aluminates, which harden very quickly, are the most important constituents of aluminous cements and high alumina blast-furnace slags, while the calcium silicates present determine the slower setting of Portland cement and blast-furnace

cements. No hydraulic binding materials are known which do not contain lime. B. W. CLARKE.

Thermal investigations on blast-furnace slags. R. GRÜN (Mitt. Forschungsinst. Hüttenzement-Ind., 1925. Reprint. **I. Latent energy of blast-furnace slags,** 5—19. **II. Latent energy of blast-furnace slags and the single components of the three-component system silica–lime–alumina,** 20—39).—The hydraulic properties of blast-furnace slag depend on the development of a vitreous product, which passes into the crystalline form when heated to about 800°, with the evolution of heat. The heat evolved during this transition is not a direct measure of the hydraulic properties of the slag cement, except with slags of the same composition, but depends on the extent of vitrification, and may therefore be used as an exact method for comparing the efficiency of various granulation processes from the point of view of the hydraulic properties of the slags. Thus, preliminary cooling of the molten slag before granulation, and overheating of the slag gives a low vitreous content and poor hydraulic properties ; air granulation, although giving less vitrification than the wet process, produces slag more active chemically and having the better hydraulic properties. Calorimetric determination of the heat of solution of slags of similar composition gives a more exact comparison of the energy content, *i.e.*, extent of vitrification, than a microscopical examination can give. The heat of hydration of a slag may be calculated from the heat of solution of the slag after deducting the heats of solution of the hydroxides produced by complete hydrolysis ; the relative proportions of these can be determined from the chemical composition of the slag. The heat of hydration, however, bears no direct relationship to the hydraulic properties of the slag. These depend chiefly on the chemical composition of the material, *e.g.*, in Portland cement the heats of solution and hydration depend chiefly on the proportion of tricalcium silicate present, while the dicalcium silicate and fluxes affect the setting or hydraulic properties of the cement. The energy contents of the single components of three- or four-component systems, such as the various types of blast-furnace slag, are most conveniently studied by the determination of the heat of solution, which gives a more accurate knowledge of the physical nature of the slag, *e.g.*, extent of devitrification, than a microscopical examination. B. W. CLARKE.

Utilisation of freshly made, hot cement. R. GRÜN and W. MUTH (Baumarkt, Leipzig, 1925, [5]. Reprint, 1 p.).—Blast-furnace cement, still hot from the grinding mills, shows no reduction in strength compared with cement which has been allowed to mature for some time before use. Similar results are to be expected with Portland cement. This disproves the view generally held that the use of freshly made, hot cement is dangerous from the strength standpoint. B. W. CLARKE.

PATENTS.

Proofing substances against insects (E.P. 247,242).—See V.

Lime-kiln (U.S.P. 1,572,156).—See VII.

Kilns for lime burning etc. (G.P. 416,143).—See XI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Melting grey cast-iron in the electric furnace. E. RICHARDS (Stahl u. Eisen, 1926, 46, 249—254).—Electric furnace cast-iron has better mechanical properties, is denser, and has a finer grained fracture than that melted in the cupola. In the basic-lined furnace the sulphur may be reduced from 0·2% to 0·05% in about 30 min., the iron is deoxidised, and the phosphorus content also lowered. The foundry is made independent of the coke supply, and the use of expensive pig iron is unnecessary. Fine scrap can be melted without loss, and large castings can be made without trouble. With the acid-lined electric furnace the advantages of purification of the metal are lost, but, on the other hand, a basic lining has a tendency to flake when subjected to temperature changes, and with intermittent operation its working life may be no greater than 8 days. Neutral lining materials were investigated. A carbon hearth, although preventing the formation of slag inclusions resulting from dissolved oxides, is strongly attacked along the slag–metal line. As the electric melting process is expensive, the " Duplex " process in which the cupola is used in combination with a basic electric furnace is recommended. The coke used in the cupola should be of low sulphur content. The chemistry of grey iron melting in a basic electric furnace is discussed with special reference to the behaviour of the manganese in the bath. Oxygen is removed by both the manganese and the carbon ; calcium carbide is formed in the neighbourhood of the electrodes, and sulphur is removed by the action of the lime in combination with the carbon and calcium carbide. The production of synthetic cast iron from small steel scrap in the basic electric furnace is described. Silicon is taken up from the basic slag or may be added more quickly as ferro-silicon. Ferromanganese is added in the ladle. Synthetic grey iron containing 2·65% of total carbon had a tensile strength of 32—35 kg./mm.^{2} and *d* 7·5. The iron was dense and free from inclusions, the graphite being finely devided in a ground-mass of pearlite and silicon-ferrite. T. H. BURNHAM.

Influence of the grain size on the magnetic properties of sheet silicon-iron. O. VON AUWERS (Wiss. Veröff. Siemens-Konz., 1925, 4, 266—273).—A more detailed account of work the results of which have been published previously (cf. B., 1926, 15). G. M. BENNETT.

Dilatometric investigation of the A3 and A4 transformations in pure iron. S. SATÔ (Sci. Rep. Tôhoku, 1925, 14, 513—527).—In the case of pure iron, during heating, the A4 transformation is accompanied by an expansion and the A3 transformation by a contraction, these changes being reversed on cooling and the ratio of the magnitudes of the change in length being about A4:A3::10:3. As the

temperature reaches 1400° an abrupt increase in length is observed due to the A4 transformation and then the rate of expansion becomes less than in the γ region and the deflection–temperature curve lies in the prolongation of that of α-iron, indicating that δ-iron is the same phase as α-iron. C. A. King.

Solidus line in the iron–carbon system. S. Kaya (Sci. Rep. Tôhoku, 1925, 14, 529—536).—The solidus line in the iron–carbon system was determined by the electrical resistance method using Swedish steels ranging from 1·60% C (solidus point 1140°) to 0·23% C (solidus point 1444°). The solidus line as determined lies between the curve as obtained by Carpenter and Keeling (B., 1904, 608) and that of Gutowsky (B., 1910, 91). The solubility limit of cementite determined from the point of intersection of the solubility and the eutectic lines was 1·7% of carbon, as at present accepted.
C. A. King.

Determination of the heat of precipitation of cementite from α- and β-martensites. M. Kawakami (Sci. Rep. Tôhoku, 1925, 14, 559—568).—The heat of precipitation of cementite from α-martensite was measured for eight kinds of carbon steel (0·3—1·32% C), by tempering a specimen in a calorimeter containing a eutectic mixture of potassium, sodium, and lithium nitrates, and maintained in a thermostat. Differentiation between α- and β-martensite was effected by tempering α-martensite below 200° and both the forms at above 300°. The heat of precipitation increases proportionally to the content of carbon and for eutectoid steel is 3·7 cal. per gram (α-martensite). The heat from α-martensite is about half as large as that from β-martensite.
C. A. King.

Influence of thermal treatment on silver alloys. L. Guillet and J. Cournot (Compt. rend., 1926, 182, 606—609).—The results of the thermal treatment of two silver–zinc and two silver–cadmium alloys containing 69·77, 62·28, 59·96, and 49·47% Ag, respectively, are recorded. On annealing the first alloy two constituents appear, one of which increases on quenching at 400°. On tempering a finely-divided precipitate appears in this constituent, accompanied by an increase in hardness and brittleness. Exactly analogous changes are shown by duralumin. The second alloy behaves in the same way. The results for the silver–cadmium alloys are less definite, though for both types of alloy the accepted phase-rule diagrams are apparently not quite correct.
S. K. Tweedy.

Hardness of cold-rolled copper. S. L. Hoyt and T. R. Schermerhorn (Inst. Metals, Mar., 1926. Advance copy, 24 pp.).—Hardness tests were carried out by the scleroscope, the Rockwell hardness tester, and by ball indentation tests on two series of cold-rolled electrolytic copper bars, originally 0·75 in. square in section, one series receiving a 2% and the other a 10% reduction in thickness per pass. The results confirm the accepted form of the relationship between the degree of working and the hardness of copper. The reversal in hardness with excessive cold working, noted by Rawdon and Mutchler (B., 1924, 260), was not found. The 10% reduction series gave a slightly greater increase in hardness than the 2% series. The hardness over the cross-section was found to be uniform for reductions of above 20%, but with small reductions in thickness the top surface was harder than the side of the bar. No definite relationship was found to exist between the hardness numbers of annealed and cold-worked copper as obtained by the three methods used. The time of loading in the ball indentation test was from one to several hours, since true equilibrium between the ball and the sample was not obtained in the standard time of 30 sec. Meyer's constant n (in the equation $P=ad^n$) decreases from about 2·4 for annealed copper to an approximately constant value of 2·0 at about 20% reduction in thickness, but as the hardening is by no means completed at this point, it follows that the constant n does not apply accurately to deformations due to cold-rolling. The increase noted in a after n has attained a constant figure is discussed, a possible explanation being that while further cold-rolling hardens the metal, the indentations made by the ball have no such effect.
A. W. Hothersall.

Soft soldering of copper. T. B. Crow (Inst. Metals, Mar., 1926. Advance copy, 14 pp.).—The interfacial effects in the soft soldering of copper were studied by the micro-examination of sections of joints prepared by soaking the ends of electrolytic copper rods in a bath of solder of eutectic composition for 10—20 min. at temperatures ranging from 237° to 497°. The solder was allowed to solidify *in situ* after adjusting the gap between the rods to the desired dimension. In the case of joints made at temperatures below 300°, a chemical reaction takes place at the copper surface which involves the production in the solid state of a structureless greyish-white band of the constituent "*H*" (CuSn). The extent of this reaction depends upon time and temperature and may be so slight as to leave traces of original surface markings upon the copper. The constituent "*H*" is soluble in excess of tin, so that increased periods of soaking do not produce thicker bands. The adhesion between the "*H*" constituent and the copper is relatively high, fracture in the tensile test never occurring so as to expose raw copper. At temperatures higher than 300° there is formed between the "*H*" and the copper a blue-mauve material which has been identified with the η phase of the bronzes (Cu_3Sn). It is weaker and more brittle than the "*H*" constituent. The increase of tensile strength of joints with diminished thickness of solder film is progressive until the film becomes discontinuous as a result of union of the two bands of "*H*" constituent across the gap. A. W. Hothersall.

Loss of zinc by brass in corrosion. G. Masing and L. Koch (Wiss. Veröff. Siemens-Konz., 1925, 4, 257—265).—Systematic experiments on the selective corrosion of brass show that the process

involves the complete solution of the brass and subsequent reprecipitation of the copper. It is due to the potential difference between brass and copper, and may therefore be regarded as an electrolytic phenomenon. G. M. BENNETT.

Brittle ranges in bronze. W. L. KENT (Inst. Metals, March, 1926. Advance copy, 8 pp).—The brittle ranges have been investigated by the Izod impact test at temperatures up to 700° on alloys, in both the cast and annealed condition, containing up to 25% Sn. The brittle copper–tin alloys containing δ in the form of $\alpha+\delta$ eutectoid lose their brittleness at 520° due to the replacement of δ by β. The β constituent so formed is soft and thus these alloys are workable above this temperature. Owing to the softness of the metal the upper limit of the range of malleability in the eutectoid alloys has not been determined, but the range extends over at least 150°. M. COOK.

Die-casting of aluminium alloys. G. MORTIMER (Inst. Metals, Mar., 1926. Advance copy, 27 pp.).—The slush, centrifugal, Cothias, gravity, and pressure methods of die-casting are reviewed. Of these the last two are most generally applicable to industry. Economic considerations are an important factor in the choice between gravity and pressure methods and the former seems more suitable for industrial requirements in Great Britain. The equipment and operation of a gravity casting installation are described. M. COOK.

Effect of artificial ageing on aged aluminium alloys. K. L. MEISSNER (Metal Ind., 1925, 26, 623—626; Chem. Zentr., 1926, I., 1029; cf. B., 1925, 321).—Two aluminium alloys, one containing copper and both of which aged at ordinary temperatures, were subjected to artificial ageing. The treatment consisted of annealing at various temperatures followed by air cooling. The most favourable annealing temperature was 125°. The physical properties of the alloy containing no copper were thereby improved in contrast to those of the copper alloy. The difference is ascribed to the inertness of the compound $CuAl_2$. L. M. CLARK.

Influence of ageing on the corrodibility of aluminium alloys. K. L. MEISSNER (Korrosion u. Metallschutz, 1925, 1, 206—208; Chem. Zentr., 1926, I., 1704).—Artificial ageing of aluminium alloys containing copper but no magnesium, *e.g.*, lautal, reduces the resistance to corrosion as measured by the oxidising salt test of Mylius (B., 1925, 552). This is probably due to the separation, during ageing, of the excess copper in a highly dispersed ultramicroscopic form. The best tensile and mechanical properties of these alloys are obtained after an ageing treatment that yields an alloy which loses 70 g. of metal per sq. m. per day in the salt test.
A. R. POWELL.

Specifications for aero-alloys. S. DANIELS (Foundry, 1925, 53, 1003—1006; Chem. Zentr., 1926, I., 1702).—Details are given of the metals and alloys used in the manufacture of light alloys for aeronautical work. The six principal alloys used are as follows: (1) 8% Cualuminium alloy, *d* 2·85, with a minimum tensile strength of 21,000 lb. per sq. in., an elongation of 1%, a Brinell hardness of 60, and a limit of proportionality of 7000 lb. per sq. in. (2) 88·5% Al, 10% Cu, 1·25% Fe, and 0·25 Mg; this alloy retains its hardness and tensile strength at high temperatures. Its ultimate strength is 25,000 lb. per sq. in., elongation 1%, and hardness 80; after ageing at 20° for 6 months these values become 27,000 lb., 0%, and 95 respectively and remain unchanged up to 260°. With suitable heat treatment the hardness of the sand-cast alloy may be increased to 120 and that of the chill-cast to 150°. The microstructure shows a network of $CuAl_2$ crossed by long needles of the iron constituent with traces of Mg_2Si. (3) 95·75% Al, 2·5% Cu, 1·25% Fe, and 0·5% Mg. This alloy is suitable for the manufacture of small parts required to withstand shock and compression. After annealing at 525° for 2 hrs., quenching in boiling water, and ageing at 150° for 2 hrs. it has a tensile strength of 31,000 lb. per sq. in., an elongation of 3%, and a hardness of 68. (4) 94% Al, 5% Cu, 1% Si, is one of the most useful alloys on account of its high tensile strength and resistance to compression; the necessary heat treatment consists of an anneal at 510° for 24—96 hrs., quenching in cold water, and ageing for 2 hrs. at 150°. (5) 93% Al, 4% Cu, 3% Si. This alloy is suitable for large castings; its microstructure consists of thin feathery crystals of $CuAl_2$, dispersed masses of the aluminium–silicon eutectic, a fair amount of needles of unknown composition, and cubes of a blue grey constituent in a ground mass of aluminium-rich solid solution. (6) 92·5% Al, 4% Cu, 2% Ni, and 1·5% Mg. The cast alloy has a tensile strength of 26,000 lb. per sq. in., an elongation of 0·5%, and a hardness of 80; after ageing for 6 months at 15—20° the corresponding values are 28,000 lb., 0·5%, and 95. The tensile strength at 260° is 23,000 lb. per sq. in. and after suitable heat-treatment may be increased to 36,000 lb. The microstructure shows $CuAl_2$, NiAl, Mg_2Si, the ternary copper–nickel–aluminium compound, and a ferruginous constituent derived from impurities in the metals used. A. R. POWELL.

Analysis of commercial magnesium. R. GUÉRIN (Ann. Chim. analyt., 1926, 8, 34—40).—Chlorine is determined colorimetrically by means of a 0·6% solution of silver nitrate if 0·01% or less is present, or gravimetrically if more than 0·01% is present. Silica is determined as in aluminium by dissolving in a mixture of equal volumes of water and a mixed acid (100 c.c. HNO_3, *d* 1·38, 300 c.c. HCl, *d* 1·18, 100 c.c. H_2SO_4, *d* 1·84, 500 c.c. of water), evaporating to dryness, treating with dilute sulphuric acid until clear, boiling, filtering, calcining the precipitate, weighing, and finally treating with hydrofluoric acid. Any lead or barium will be partially retained by the silica as sulphate. Iron is determined by titration with potassium permanganate. Manganese is determined colorimetrically as in steels, by means of silver nitrate and ammonium

persulphate. For copper, if 0·2% or less is present, the blue colour developed with ammonia is compared with those from type samples; if more than 0·2%, after separation of silica an electrometric method is used in the cold with Hollard's electrodes. Lead is determined as sulphate. For aluminium the hydrochloric acid solution is treated with bromine water, and after boiling, with saturated ammonium chloride, then with ammonia until alkaline, whereby aluminium, iron, and manganese are precipitated. The precipitate must be freed from any magnesium oxide; the oxides are weighed and the aluminium determined by difference. Zinc is determined by electrolysis of a solution containing the zinc and magnesium after separation of copper. Calcium is separated from magnesium by a method depending on the different solubilities of the oxalates.

D. G. Hewer.

Interpretation of the macrostructure of cast metals. R. Genders (Inst. Metals, Mar., 1926. Advance copy, 21 pp.).—In steel ingots the commonly known regular distribution of the different types of macrostructure appears to be due largely to the low rate of solidification resulting from the relatively low conductivity of the metal. Ferrous alloys will permit of slow casting with a superheat of less than 50°, and under such conditions a maximum of equiaxed crystals with a minimum of columnar crystals is produced. Where the thermal conductivity of a cast alloy is high, however, as in non-ferrous alloys, solidification is rapid, occurring concurrently with pouring, which does not allow the turbulence of the metal during pouring and the path of the stream to be smoothed out. These facts are directly reflected in the macrostructure Observations on the distribution of metal when cast in a mould were made by pouring a molten "red" brass (90% Cu) followed immediately by a white nickel-silver of the same order of conductivity. The character of the macrostructure of an alloy is influenced by the range between liquidus and solidus. Ingots of 70 : 30 brass, which has a relatively long freezing range, may show a proportion of equiaxed crystals, whilst a similarly cast ingot of 60 : 40 brass, which has a very short range, consists entirely of columnar crystals.

C. A. King.

Relation between the equilibrium diagram and the magnetic susceptibility in binary alloys. H. Endo (Sci. Rep. Tôhoku, 1925, 14, 479—512).—The magnetic susceptibility-concentration curve becomes a straight line in the case of alloys consisting of a mechanical mixture of two constituents, but when alloys form a solid solution the same curve takes the form of a curved line. The magnetic susceptibility was determined in relation to the equilibrium diagram in the systems, Cu-Sn, Cu-Zn, Zn-Sn, Pb-Te, Bi-Pb, Sb-Sn, Sb-Cu, Bi-Zn, Cu-Mn, Bi-Sn, Sb-Pb, Bi-Te, the equilibrium diagrams of the last three systems being revised on the basis of the magnetic analysis.

C. A. King.

Corrosion of an ancient tin specimen. C. O. Bannister (Inst. Metals, Mar., 1926. Advance copy, 2 pp.).—Examination of the corroded layer of a tin scabbard end of mediæval or possibly Roman period showed that the mechanism of corrosion of tin comprises the formation of hydrated stannous oxide and the subsequent oxidation and dehydration of this compound to stannic oxide.

C. A. King.

Separation of tin and antimony, especially from tin-antimony-lead alloys, by the dry method. W. Lidle (Metall u. Erz, 1926, 23, 5—10; Chem. Zentr., 1926, I., 1862).—Tin and antimony cannot be separated from lead by addition of alkali or alkaline-earth metals to the molten alloy, followed by liquation; *e.g.*, if magnesium is added, mixed crystals of $SnMg_2$ and Sb_2Mg_3 are produced which form a eutectic with the other constituents and this liquates with the lead. Small quantities of antimony may be separated from tin-lead alloys by addition of a tin-iron alloy which will remove up to 10% of its weight of antimony. Addition of sulphur to the molten alloy removes first tin, then lead, but much of the latter is converted into sulphide before all the tin is removed as sulphide. Thus, an alloy containing 40% Sn, 50% Pb, and 10% Sb, after treatment with sulphur, gave a metallic regulus containing 98% of the antimony, 62·6% of the lead, and 23·7% of the tin in the original alloy.

A. R. Powell.

Striation due to working or to corrosion in microscopical metallography. Mode of action of etching reagents. A. M. Portevin (Inst. Metals, Mar., 1926. Advance copy 6 pp.).—The latent scratches produced during polishing a metal and developed by etching are often visible only on certain grains, at the boundaries of which they stop, to reappear again on other crystals having a similar orientation. This is particularly marked in the case of the aluminium-copper solid solution with 2·2%Al after etching with ammonium persulphate; the surface becomes covered with big lines in the same general direction caused by fine markings or striations. The intensity and direction of striation changes on crossing a twin and the striæ bear a close relationship to etching figures, being merely etching figures on a crystalline surface the lattice of which has been disturbed by a permanent deformation set up by the scratch. In the case of α-iron, regions deformed by cold work such as scratching with a pin or squeezing in a vice show characteristic markings in the form of a silhouette after etching with copper ammonium chloride and nitric acid. After removal of a pin-scratch on the metal by polishing, deep etching for etching figures reveals the path of the scratch by the development of striations which deform the etching figures. After annealing metal which has been scratched, new grains develop from the regions where the lattice has been deformed and twins are formed in the parts that have suffered proportional translation.

A. R. Powell.

Behaviour of some metals and alloys with plastilin and free sulphur. O. Bauer and H. Arndt (Z. Metallk., 1926, 18, 85—88).—Copper becomes slowly converted into sulphide in contact

with plastilin (containing 40% of zinc oleate, 18% of wax and oil, 15% of kaolin, and 23% of sulphur), the depth of penetration increasing with the time to a maximum of about 0·5 mm.; further action is then prevented by the copper sulphide layer formed. The action is more rapid the higher the temperature, but the maximum depth of penetration is the same. Brass and bronze are hardly affected even by prolonged contact with plastilin, and aluminium, zinc, and tin are only slightly discoloured. Aluminium, zinc, tin, and especially manganese prevent the action of plastilin on copper and appreciably reduce the severity of the action of molten sulphur on copper. An alloy of 33·3% Mn and 66·7% Cu is not attacked by sulphur at 400° even after 5 hrs., whereas pure copper loses 2 g. per sq. dm. in the same time. Lead is almost as severely attacked by plastilin as is copper. A. R. POWELL.

Cold-deformation or corrosion fringes. A. PORTEVIN (Compt. rend., 1926, **182**, 523—525).—A study has been made of the effects of chemical reagents on the polished surfaces of metals subjected to cold deformation, and in particular of the alternate bright and dark striations or "fringes" produced. These were of two distinct types, namely, those which showed distinct alternating fringes and channels, and those in which the corrosion patterns were modified. These were studied for copper and α-iron respectively. It is concluded that the patterns are characteristic of a permanent crystalline state, but that where the deformations follow directions not strictly parallel, the consequent fineness of structure and distortion of the lattice give the appearance of an amorphous layer.

J. GRANT.

Volumetric determination of uranium, vanadium, copper, and iron in uranium ores. A. S. RUSSELL (J.S.C.I., 1926, **45**, 57—60 T).—Methods for determining volumetrically some or all of the elements named in solution in sulphuric acid have been worked out, and applied principally to the determination of these elements in uranium ores. Uranous sulphate is used as a new reducing agent, and previous work on liquid zinc amalgam as a reducing agent, and of diphenylamine as an indicator of vanadic acid, has been made use of. The results for the vanadium, uranium, and iron contents of carnotite; the uranium, copper, and iron contents of torbernite; and the uranium and iron contents of pitchblende, thorianite, autunite, and other ores agree satisfactorily with those obtained gravimetrically. A modification of the usual method for determining quadrivalent uranium in the oxide U_3O_8 is suggested.

"Atomised" coal for smelting non-ferrous metals. R. BLACK and C. L. SHAFER (Metal Ind., 1925, **23**, 403—404; Chem. Zentr., 1926, I, 1030—1031).—The use of "atomised" coal (all of which passes through a 200-mesh sieve) instead of coal "dust" (75% through the 200-mesh sieve) brings definite advantages. Starting from cold, combustion is complete after a period of 3—4 min.; the ash melts and is found as slag on the walls of the furnace, the outside of the crucible, and the surface of the charge. Weekly removal of this loose and friable slag suffices. The preparation of the carbon as well as the furnaces suitable for its use, and the cost are discussed. L. M. CLARK.

α-Phase boundary in the copper–tin system. D. STOCKDALE (J. Inst. Metals, 1925, **34**, 111—124).—See B., 1925, 809.

Constitution of alloys of aluminium, copper, and zinc. D. HANSON and M. L. V. GAYLER (J. Inst. Metals, 1925, **34**, 125—170).—See B., 1925, 809.

Constitution of zinc–copper alloys containing 45—65% Cu. M. L. V. GAYLER (J. Inst. Metals, 1925, **34**, 235—244).—See B., 1925, 808.

β-Transformation in copper–zinc alloys. J. L. HAUGHTON and W. T. GRIFFITHS (J. Inst. Metals, 1925, **34**, 245—260).—See B., 1925, 808.

Physical properties of the copper–cadmium alloys rich in cadmium. C. H. M. JENKINS (J. Inst. Metals, 1925, **34**, 85—102).—See B., 1925, 764.

Zinc–cadmium alloys. R. B. DEELEY (J. Inst. Metals, 1925, **34**, 193—206).—See B., 1925, 810.

Properties of some aluminium alloys. H. HYMAN (J. Inst. Metals, 1925, **34**, 207—234).—See B., 1925, 764.

Passification and scale resistance in relation to the corrosion of aluminium alloys. L. H. CALLENDAR (J. Inst. Metals, 1925, **34**, 57—84).—See B., 1925, 809.

Influence of pouring temperature and mould temperature on the properties of a lead-base anti-friction alloy. O. W. ELLIS (J. Inst. Metals, 1925, **34**, 301—322).—See B., 1925, 809.

Colloidal separations in alloys. J. H. ANDREW and R. HAY (J. Inst. Metals, 1925, **34**, 185—192).—See B., 1925, 809.

Thermal conductivities of industrial non-ferrous alloys. J. W. DONALDSON (J. Inst. Metals, 1925, **34**, 43—56).—See B., 1925, 810.

The high-temperature tensile curve. D. H. INGALL (J. Inst. Metals, 1925, **34**, 171—184).—See B., 1925, 764.

Influence of time factor on tensile tests as high temperatures. J. S. BROWN (J. Inst. Metalt, 1925, **34**, 21—42).—See B., 1925, 810.

Effect of temperature on the behaviour of metals and alloys in the notched-bar impact test. R. H. GREAVES and J. A. JONES (J. Inst. Metals, 1925, **34**, 85—102).—See B., 1925, 765.

Electrodeposition of zinc from sulphate solutions.—A. L. MARSHALL (Trans. Faraday Soc., 1925, **21**, 297—318).—See B., 1925, 636.

Patents.

Production of high-tensile steel. R. W. James. From Eisenwerk-Ges. Maximilianshütte (E.P. 247,276, 11.11.24).—Steel having high resistance to wear combined with great toughness, suitable for rails, tyres, and the like, contains 0·3—0·7% Si and 1·25—2·5% Mn, the combined amount of silicon and carbon being not greater than 0·7% and the proportions of these two elements being interdependent, the silicon rising from 0·30% to 0·50% as the carbon falls from 0·40% to 0·20%. By this means the formation of brittle iron carbide (Fe_3C) is retarded, Fe_3Si or Fe_3C/Si being predominantly formed, and as large a quantity of the carbon as possible is combined with manganese in the form of Mn_3C or Mn_3C/Si. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 131,980.)
T. H. Burnham.

Desulphurisation and concentration of sulphide iron ores. W. S. Millar (E.P. 247,471, 12.8.25. Addn. to 236,256; B., 1925, 725).—The ore is subjected to the action of sulphur dioxide gas of at least 50% purity in a furnace, *e.g.*, a mechanical roasting furnace, at a gradually rising temperature, with or without the admission of steam. Part of the sulphur is first driven off leaving the ore with the approximate composition FeS, and then, without fusion, the remainder of the sulphur is driven off as free sulphur at a temperature not exceeding 950°, leaving a porous, highly magnetic product. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 236,256). T. H. Burnham.

Electrothermic refining of metals [steel]. J. L. Dixon, Assr. to Pittsburgh Research Corp. (U.S.P. 1,569,464, 12.1.26. Appl., 5.9.19).—Steel is refined in an electric arc furnace having a number of electrodes, low-voltage current being supplied between one pair of electrodes and relatively high-voltage current between other electrodes. J. S. G. Thomas.

Rust-proofing bath for iron and steel. J. H. Gravell (U.S.P. 1,572,354, 9.2.26. Appl., 9.5.22. Renewed 3.11.25).—A bath for treating iron in order to prevent rusting comprises a solution of phosphoric acid and sodium dihydrogen phosphate. The latter serves to neutralise the pickling acid without forming a sludge. A. R. Powell.

Manufacture of steel. Soc. des Prod. Métallurgiques Constant-Bruzac (F.P. 594,547, 19.5.24).—The finely divided ore is heated to the temperature required for reduction and treated with carbon moxoxide until the iron is saturated with carbon. The product is separated from gangue either before or after melting in the absence of air, and is treated with lime, ferric oxide, or the like to remove deleterious metalloids. The carbon content is then reduced in the usual way until steel of the required composition is produced, oxygen being subsequently removed by the addition of manganese; or alternatively, the iron saturated with carbon is melted with the necessary amount of iron free from carbon.
L. M. Clark.

Steels. M. Baeke (F.P. 595,530, 20.3.25).—Steels, which are very resistant to acids and alkalis, and are not affected by air, contain Cr 8—25%, Mn 2—25%, and C 0·4—1·5%. L. M. Clark.

Reduction of poor oolitic iron ores. J. Bing (G.P. 422,030, 25.7.23).—Poor oolitic iron ores are reduced at high temperatures with the hot carbon monoxide produced as a by-product in the manufacture of calcium carbide in the electric furnace. The product is smelted in the electric, cupola, or open-hearth furnace. A. R. Powell.

Reduction of ores, especially iron ores. Gewerkschaft Alflen VII (G.P. 423,072, 28.3.24).—Iron ores are reduced by heating with semi-coke in revolving or reverberatory furnaces. The reducing power of semi-coke is greater than that of metallurgical coke and resembles that of charcoal. A. R. Powell.

Reduction of iron oxide ores and residues with reducing gases. R. Schenck and T. Thorsell (G.P. 423,501, 23.6.22).—Iron oxide ores and residues are reduced with gases at a temperature below the sintering temperature. The composition of the reducing gas mixture and the partial pressure of the reducing gases and their combustion products are so adjusted in relation to the temperature at which the reduction is carried out that the reduction is carried to the desired stage. With suitable composition of the reducing gases it is possible to produce iron free from carbon or containing more or less carbon, iron carbide, or solid solutions of iron carbide in iron. A. R. Powell.

Spectroscopic testing of the metal bath in the refining of iron in the electric furnace. Felten u. Guilleaume Carlswerk A.-G. (G.P. 423,436, 27.6.24).—An arc is struck between one part of the metal bath and a secondary electrode of known spectrum and the vapour so produced is observed through a spectroscope. A. R. Powell.

Manufacture of steel or malleable iron. Linke-Hofmann-Lauchhammer A.-G., Assees. of E. Wiegand (G.P. 423,475, 17.12.24).—Scrap-iron is heated to a high temperature by external heat, and oxygen or oxidising substances are introduced so that partial combustion of the iron takes place while the heat thus generated fuses the remainder of the scrap; subsequently the iron oxide formed in the first stage is reduced in the usual way. A. R. Powell.

Case-hardening. W. J. Merten (U.S.P., 1,571,289, 2.2.26. Appl., 2.12.20).—Case-hardening is effected by means of cyanogen gases produced by heating a cyanide, and the molten cyanide is agitated by bubbling the unabsorbed gases through it.
M. Cook.

Direct case-hardening of iron articles in molten cyanide baths. Allgem. Elektricitäts-Ges., Assees. of H. Blomberg (G.P. 422,305, 13.3.25).

—To increase the life of the cyanide bath small compressed pieces of charcoal are added to prevent the formation of cyanate. A. R. POWELL.

Recovery of nickel or nickel–iron alloys from nickeliferous rolling-mill scale and similar residues. M. STERN (G.P. 422,325, 26.6.18).—Rolling-mill scale and similar residues containing nickel are smelted with iron in a cupola or on the hearth of a reverberatory furnace in such a way that the nickel is precipitated from the fused oxides by the metallic iron. A. R. POWELL.

Metallurgical process [for treating copper ores]. W. E. GREENAWALT (U.S.P. 1,572,114–5, 9.2.26. Appl., [A] 2.9.24, [B] 1.12.24).—(A) Oxidised copper ore or roasted concentrate is leached with an acid solution and the resulting copper solution is subjected alternately to the action of a reducing agent (*e.g.*, roaster gases containing sulphur dioxide) and to electrolysis to deposit the copper. The cycle is repeated until the solution becomes foul, when a quantity of the liquid is diverted to a separate circuit where it is partially neutralised and the same cycle of operations is repeated on this solution until only a small amount of copper is left. This is precipitated with hydrogen sulphide and the solution is treated for the separation of nickel, cobalt, and zinc, while the copper sulphide is returned to the circuit. (B) The copper solution obtained by leaching copper ores with acid is electrolysed in a series of units each comprising a feed tank, a reducer tank, and an electrolytic cell, and a substantially constant flow of electrolyte in a closed circuit is maintained in each unit, excess electrolyte being by-passed occasionally through the feed tanks. A. R. POWELL.

Pickling [bath for metals]. H. S. MARSH and R. S. COCHRAN, Assrs. to AMER. COPPERAS CO. (U.S.P. 1,572,155, 9.2.26. Appl., 14.11.24).—A pickling solution for cleaning metal articles is saturated with hydrogen sulphide. A. R. POWELL.

Treating mixed lead–zinc sulphide ores. N. C. CHRISTENSEN (U.S.P. 1,572,268, 9.2.26. Appl., 31.12.23).—By treating the finely ground ore with acid, concentrated brine, the lead and part of the zinc are dissolved as chlorides. The evolved hydrogen sulphide is absorbed by lime, and the calcium hydrosulphide formed is used to precipitate first the lead, and then the zinc. A. GEAKE.

Recovery of lead and zinc from ores. H. and P. FAIVRE (F.P. 596,147, 3.7.24).—Finely divided oxidised lead ores are treated with boiling ammonium chloride solution, and the lead ammonium chloride solution so formed is separated from the gangue and cooled, whereby the bulk of the double salt crystallises out and the solution can be used again. Similarly oxidised zinc ores are leached with boiling ammonium sulphate solution which converts any lead into sulphate and dissolves the zinc as zinc ammonium sulphate, which is recovered by cooling the filtered solution. A. R. POWELL.

Recovery of lead and silver from sulphide ores and metallurgical products. H. HEY (U.S.P. 1,572,388, 9.2.26. Appl., 20.1.21).—Sulphide ores are heated in the presence of gaseous hydrogen chloride under non-oxidising conditions to convert the lead and silver present into chlorides while inhibiting the conversion of the other sulphides in the ore. The lead and silver chlorides are then leached out with suitable solvents.
T. H. BURNHAM.

[Aluminium] alloy. R. L. JOHNSTON, Assr. to ALUMINUM DIE-CASTING CORP. (U.S.P. 1,572,357, 9.2.26. Appl., 26.12.22).—An aluminium alloy containing 5—10% Sn and a substantial amount of silicon is claimed. A. R. POWELL.

Refractory lining for crucibles [for aluminium alloys]. F. C. FRARY, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,572,475, 9.2.26. Appl., 15.1.24).—A crucible or ladle for handling aluminium alloy at high temperatures consists of a steel shell lined with finely ground alumina bonded with sodium silicate.
A. R. POWELL.

Aluminium alloy for motor pistons. ALUMINIUM-INDUSTRIE A.-G. (Swiss P. 112,246, 25.9.24).—An aluminium alloy for the manufacture of pistons for motor-cars, contains 8—17% Cu, 1—3% Fe, and up to 2% Mg, Mn, or similar hardening element. The castings are either cooled very slowly or annealed below 350° so as to prevent subsequent changes in the hardness and shape. A. R. POWELL.

Apparatus for refining aluminium. F. WINZENRIED and R. TIMENS (Swiss P. 98,838, 3.6.21).—An apparatus for refining aluminium consists of a tilting furnace with a charging opening in the roof. The opening is constricted towards the lower end and bent upwards. The whole is covered with a hood carrying a tubular flue which is built into the furnace lining and ends in a retort also built into the lining. The retort serves for the vaporisation of reagents which are passed through the tube into the aluminium during the refining process. A. R. POWELL.

Alloy. E. R. CROSBY. Assr. to ALUMINUM MANUFACTURERS, INC. (U.S.P. 1,572,382, 9.2.26. Appl., 7.7.21).—An aluminium alloy contains 3—8% Si and up to 5% Sn. T. H. BURNHAM.

Production of [refractory] metals. J. W. MARDEN and C. C. VAN VOORHIS, Assrs. to WESTINGHOUSE LAMP CO. (U.S.P. 1,573,083, 16.2.26. Appl., 13.6.21).—Refractory metals are produced by heating their oxides with an alkali metal and a halide of another alkali metal or of an alkaline-earth metal. A. R. POWELL.

Concentration of oxidised ores. S. CROASDALE (U.S.P. 1,573,226, 16.2.26. Appl., 8.1.23).—Ores containing oxidised minerals may be concentrated by oil-flotation by treating the ore pulp with calcium pentasulphide, a frothing agent, and a relatively large amount of a hydrocarbon mixture containing a large proportion of olefines. No visible film of

metal sulphide is formed on the surface of the mineral particles during treatment.
A. R. POWELL.

Recovering metals from slags. H. ZANICOLI (F.P. 594,928, 23.9.25).—The molten slag is heated to a high temperature and treated with reducing vapours, such as those of heavy petroleum hydrocarbons, in one chamber of a container divided by a bridge. The greater part of the heavy metal silicates present is reduced to the metal, which flows over the bridge and is mechanically separated.
L. M. CLARK.

Bearing metal alloys with a bronze foundation. T. GOLDSCHMIDT A.-G. (G.P. 420,068, 6.7.21; F.P. 594,914, 10.3.25).—A bearing metal has the composition Pb 12—25%, Ni 2·5—5%, Sn 2—10%, and the remainder copper. Part of the tin may be replaced by antimony and zinc. The addition of a small quantity of phosphor-copper as a deoxidising agent often improves the casting. L. M. CLARK.

Lead-zinc-antimony bearing metal. F. DOEBLIN (G.P. 423,450, 15.5.23. Addn. to 367,978 and 382,545; B., 1923, 408; 1924, 388).—Part of the lead, specified in the previous patents, is replaced by an equal weight of copper, up to 10% of the alloy.
A. R. POWELL.

Alloy for soldering lead alloys to iron, bronze, and the like. METALL-VERARBEITUNGSGES. M.B.H. (G.P. 423,293, 28.8.24).—A solder for joining lead bearing metal alloy containing alkali or alkaline-earth metals to iron or copper alloy bases consists of lead with the addition of other metals which lower its m.p. and render it plastic between 160° and 220°. A suitable alloy comprises 52·5% Pb, 42·5% Sn, and 5% Zn. A. R. POWELL.

Coatings of fusible substances [metals]. M. U. SCHOOP (Swiss P. 108,499, 10.3.24).—The coating substance is melted and sprayed from a jet in such a way that the stream is broken up into fine droplets. This may be effected by means of shaking devices inside the orifice of the jet, by spraying the substance from two jets inclined at an angle towards each other, or by using pulsating pressures to force the substance through the jet. The coatings so obtained are less brittle and softer than those produced by the ordinary metal spraying process. A. R. POWELL.

[Ore] separating process. R. ELLIS, Assr. to ELLIS FLOTATION CO. (Reissue 16,279, 2.3.26, of U.S.P. 1,425,186, 8.8.22. Appl., 1.8.24).—See B., 1922, 765 A.

Osmium alloy. E. HAAGN, Assr. to W. C. HERAEUS G.M.B.H. (U.S.P. 1,574,966, 2.3.26. Appl., 29.3.21).—See G.P. 350,703; B., 1922, 505 A.

Recovering light metals from scrap and the like. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. BECK (U.S.P. 1,576,080, 9.3.26. Appl., 11.4.24). —See E.P. 219,287; B., 1925, 75.

Vanadium compounds (F.P. 598,315).—See VII.

Sulphur dioxide and sulphuric acid from sulphur-bearing slag (G.P. 423,640).—See VII.

Preparing calcium amalgams (G.P. 420,721).—See XI.

XI.—ELECTROTECHNICS.

Insulating oils. H. STÄGER (Z. angew. Chem., 1926, 39, 308—316).—The dielectric constant of insulating oils increases rapidly with rise of temperature so that breakdown of an insulating layer of oil may be due to local overheating. A very small proportion of moisture rapidly decreases the resistance of oil but further addition of moisture has little effect. The most serious cause of breakdown is the presence of acidic constituents produced by atmospheric oxidation, and the various methods proposed for determining the rate of oxidation are discussed and criticised at length. A. R. POWELL.

Influence of exterior temperature on temperature of standard pyrometric electric lamps. RIBAUD.—See I.

Products of decomposition of mineral oil by electric arc. EVERS.—See II.

PATENTS.

Electric resistance furnaces. G. E. TAYLOR, and ELECTRIC FURNACE CO., LTD. (E.P. 247,254, 20.7.25).—The cross sections and lengths of the resistors and the thickness of the refractory walls and roof of a furnace supplied with two-phase or three-phase current are proportioned so that the full load taken by the furnace on heating up is as nearly as possible respectively twice or three times the power required to balance the loss by radiation and to maintain the furnace at the desired temperature.
J. S. G. THOMAS.

Electric-arc furnace. J. L. DIXON, Assr. to PITTSBURGH RESEARCH CORP. (U.S.P. 1,569,463, 12.1.26. Appl., 5.9.19).—An electric furnace is provided with three upper electrodes and a transformer of three-phase alternating currents having unequal transformer ratios in the several phases and supplying the electrodes with currents at different voltages. J. S. G. THOMAS.

Kiln [with electric heating for lime burning etc.]. SIEMENS U. HALSKE A.-G., Assees. of R. GROSS and M. STADLHUBER (G.P. 416,143, 22.2.22).—A kiln for burning lime, magnesite, cement, or the like consists of a circular rotating horizontal hearth covered with a dome and sealed by a sand or liquid seal. The material to be treated is introduced on to the periphery of the hearth, and is heated by several electric arcs arranged around the hearth or by radial resistances. The heat-radiating surface may be increased by providing partitions between the arcs or resistances. At intervals the heated material is impelled to the centre of the hearth, where it is withdrawn. The inlet and outlet valves and the rakes for impelling the material towards the centre

of the hearth may be coupled mechanically, so that they work in unison. T. S. Wheeler.

[Coating electrodes of] discharge tubes, especially low-voltage glow lamps. F. Skaupy (G.P. 414,517, 3.5.22).—Electrodes for use in discharge lamps are formed of a cheap metal, such as iron, and, before or during the manufacture of the lamp, are coated with a solution or a suspension of the azide of a metal of good electron-emitting properties. An alkaline-earth azide such as barium azide is suitable, or a mixture of azides may be used. The lamp is heated during evacuation either externally or by passing a current through it, and the azide decomposes forming the corresponding metal, and nitrogen, which is pumped off. Alternatively the decomposition may be accomplished while the lamp is filled with a suitable inert gas. The alloy or coating formed on the surface of the electrodes increases the life of the lamp and enables a good light to be obtained from it even on a low voltage. T. S. Wheeler.

Preparation of high-percentage calcium amalgams by electrolysis. A. Eilert (G.P. 420,721, 21.9.24).—In the preparation of high-percentage calcium amalgams by electrolysis the mercury used as cathode and the vessel in which the electrolysis is performed are heated before introducing the electrolyte; the electrolyte is freed and kept free from suspended particles, such as undissolved calcium hydroxide, and the amalgam is compressed from time to time so that proper contact may be kept between the mercury and the lead bringing in the current. L. M. Clark.

Protecting gas-absorbing substances [in electric cells] from penetration by liquid. R. Oppenheim, Assr. to Soc. Anon. le Carbone (U.S.P. 1,574,844, 2.3.26. Appl., 26.4.23).—See E.P. 206,471; B., 1924, 986.

Electric battery. R. Oppenheim, Assr. to Soc. Anon. le Carbone (U.S.P. 1,574,845, 2.3.26. Appl., 6.9.24).—See E.P. 236,884; B., 1925, 813.

Process for manufacturing a lead accumulator. K. Kawakami (U.S.P. 1,575,167, 2.3.26. Appl., 3.10.21).—See E.P. 193,087; B., 1923, 363 a.

Cooling the electrodes in ozonisers. R. Graef, Assr. to A.-G. für Ozon Ind. (U.S.P. 1,575,049, 2.3.26. Appl., 10.2.25).—See E.P. 241,125; B., 1925, 963.

See also pages 319, **Sulphuric acid** (G.P. 421,786). 320, **Removing arsenic from burner gases** (G.P. 423,657). 321, **Gas mixtures for ammonia synthesis** (F.P. 596,714). 322, **Barium, strontium, and calcium peroxides** (G.P. 422,531); **Alkaline-earth nitrides** (G.P. 423,348); **Corundum** (G.P. 422,105); **Magnesium hydroxide** (Nor. P. 39,564). 339, **Flour-bleaching gas** (E.P. 246,979).

XII.—FATS; OILS; WAXES.

Composition of olive oil. K. Täufel and J. G. Sarria (Anal. Fís. Quím., 1926, 24, 25—40).—The following data were obtained for a Spanish olive oil: $d^{18.5}$ 0·914, oleorefractometer reading at 25° 61·9, acid value, 0·62, saponif. value 192·6, iodine value (Winkler's method) 82·7, Reichert–Meissl value 0·13, Hehner value 95, m.p. of fatty acids 28·2°. It contained stearic acid 2·27%, palmitic acid 7·55%, oleic acid 83·94, linoleic acid 0·51%, glycerol (calc.) 4·44%, and unsaponifiable matter 0·79%. Triolein and α-palmitodiolein were isolated. Arachidic acid was absent. G. W. Robinson.

Surface properties of soap solutions. Structure of the fluid surface. J. F. Carrière (Chem. Weekblad, 1926, 23, 118—126; cf. B., 1923, 563).—The theory is put forward that the surfaces of soap solutions are composed of molecules of the fatty acids, oriented according to the theories of Langmuir and Harkins; experiments in support of this view are described. Thus the motion of camphor particles on the surface of water, inhibited by the presence of small quantities of sodium oleate, is momentarily re-induced by passing ammonia over the surface of the liquid; sulphur dioxide has no such effect. So also extraction of a soap solution with neutral oil increases the alkalinity of the solution. A previous experiment has been repeated with air free from carbon dioxide, and it is found that the froth carried over from the soap solution contains free fatty acid, whilst the residue is more strongly alkaline than before. The surface tension–concentration curves of Lascaray (B., 1924, 432), Walker, (B., 1921, 856 a), and others are discussed, and the theories previously put forward shown to be untenable. S. I. Levy.

See also A., Mar., 268, **Saturated acids of highest m.p. from arachis oil** (Holde and Godbole). 269, **Additive products of iodine bromide and hypoiodous acid with unsaturated compounds** (Holde and Gorgas); **Fractional dissolution in alcohol of zinc salts of liquid acids from linseed oil** (Agde); **Octadecenoic acids** (Grün and Czerny). 286, **Naphthenic acids** (Naphtali).

Converting fatty acids into hydrocarbons. Stadnikov and Ivanovski.—See II.

Ajowan and mohua cakes as fertilisers. Rege.—See XVI.

Patents.

Separating glycerides from fats and oils. Allgem. Ges. f. Chem. Ind. m.b.H. (F.P. 595,250, 17.3.25).—Saturated or nearly saturated glycerides, *e.g.*, those containing oleic acid, are separated from less saturated glycerides by suitable solvents, *e.g.*, liquid sulphur dioxide. A. Geake.

Continuous production of fatty acids, glycerin, and ammonium sulphate. V. Graubner (G.P. 421,438, 8.6.24).—An emulsion of neutral fat and

ammonia, or a mixture of the fat with gaseous, liquid, or dissolved ammonia, is run continuously into a large quantity of hot ready-formed ammonium soap. A corresponding amount overflows from the vessel and is decomposed with sulphuric acid or sulphur dioxide. The ammonium soap is preferably under a slight pressure. A. GEAKE.

Obtaining fat from fat-bearing vegetable rinds. CHEM. FABR. GRIESHEIM-ELEKTRON (Swiss P. 111,359, 5.5.24).—The rinds are mechanically disintegrated and extracted with a fat solvent, *e.g.*, trichloroethylene. A. GEAKE.

Hydrogenation of oils, fats, and fatty acids with carbon as carrier for the metallic catalyst. N. V. ALGEM. NORIT MAATSCHAPPIJ (Dutch P. 12,851, 14.11.21).—Active or decolorising vegetable charcoal, freed from mineral constituents, and retaining its structure, is used as a carrier for the catalyst; it takes up more of this than kieselguhr or the like. A. GEAKE.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Determination of the covering power, opacity, and grain size of paints. W. REGLIN (Z. angew. Chem., 1926, 39, 334—335).—A simple apparatus for use in determining the covering power, opacity, and grain size of paints and the evenness of paint films comprises a rectangular box containing a 32–c.p. electric lamp and fitted with a glass lid on which transparent films of gelatin-coated paper may be fixed. The paint may be spread on the film by means of a brush or by spraying, and the irregularities of distribution are readily apparent when the apparatus is transferred to a dark room and the lamp in the box switched on. The size of the individual particles of pigment may then be measured by means of a microscope. The films produced on gelatin-coated paper are in every way similar to corresponding films on metallic surfaces, so that this apparatus permits deductions to be drawn as to the suitability of a paint for rust-protecting coatings. A. R. POWELL.

Use of tung oil. H. KÖLLN (Farben-Ztg., 1926, 31, 920—921).—An account of the difficulties encountered in the use of tung oil in varnishes, the value of various incorporated resins—synthetic and natural—and a general discussion of the attempts made to solve the two main problems arising in practice, *i.e.*, avoidance of the irregularity in the drying of tung oil of normal viscosity and complete control of the consistency of the oil in the process of thickening. S. S. WOOLF.

Softening point of resins. W. NAGEL (Wiss. Veröff. Siemens-Konz., 1925, 4, 321—323).—A modification of the Krämer–Sarnow method for determining the softening point of pitches is described which makes it suitable for work with resins. The finely powdered material is pressed down in a small tube with a somewhat constricted but open lower end, and 5 g. of mercury are placed on it. The tube is then heated in an air-jacket in a glycerin bath and the temperature observed at which the mercury falls through the resin. The results are consistent to about 5° or 10°. Values for a number of resins are recorded. G. M. BENNETT.

Trichromatic colorimeter. Criticism of monochromatic-plus-white method of colorimetry. New method of colorimetry. GUILD.—See I.

PATENTS.

Preparation of pure *blanc fixe* and sodium thiosulphate. CHEM. FABR. GRÜNAU, LANDSHOFF & MEYER (G.P. 420,251, 10.2.24).—A solution of sodium sulphate is added to barium sulphide and air is led through until the product is colourless. Sulphur dioxide is then passed into the heated mixture until the solution has a faintly acid reaction. By this method, *blanc fixe* free from sulphur is obtained. L. M. CLARK

Preparing a painting material of powder form for use with either water or oil. M. TSUBATA (U.S.P. 1,574,854, 2.3.26. Appl., 24.7.23).—See E.P. 224,273; B., 1925, 45.

Manufacture of printing colours or ink. H. CAJAR (U.S.P. 1,576,408, 9.3.26. Appl., 5.12.23).—See E.P. 219,562; B., 1924, 797.

Manufacturing linoleum. G. DUBSKY (U.S.P. 1,576,181, 9.3.26. Appl., 14.7.25).—See E.P. 235,590; B., 1926, 202.

Proofing substances against insects (E.P. 247,242).—See V.

Coloured cellulose solutions (E.P. 247,288).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Crêpe rubber prepared with different reagents. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1926, 8, 77—79; cf. B., 1926, 68, 288).—Crêpe rubber prepared with the use of *p*-nitrophenol is not yellower in colour than much commercial "pale crêpe" rubber. As with smoked sheet rubber (*loc. cit.*) the joint use of *p*-nitrophenol and sodium silicofluoride tends to eliminate, by compensation, the effect of each of these on the rate of vulcanisation. D. F. TWISS.

Mastication [of rubber]. E. A. HAUSER and H. DANNENBERG (Kautschuk, 1925, Nov., 6—8).—The "two-phase" theory of the structure of rubber suffices to explain the phenomena of the mastication process; *e.g.*, the "recovery" of rubber on storage after mastication is attributed to globules which during the milling operation have not lost the whole of their less viscous interior, once more becoming closed, thereby restoring part of the elastic character to the material. The aggregation theory of Le Blanc and Kröger (B., 1925, 932), which is particularly applicable to the vulcanisation process, is not incompatible with the two-phase theory (cf. Hauser, B., 1925, 514). D. F. TWISS.

Structure of stretched rubber. E. A. Hauser and H. Mark (Kautschuk, 1925, Dec., 10—11). In the X-ray investigation of rubber the position of the interference spots, within an accuracy of 1%, is independent of the stretch whilst their intensity is approximately proportional to it; increase in the degree of extension also does not affect the position of the amorphous ring but decreases its intensity. When rubber is stretched a proportion of a crystalline phase results, the axes of the crystals lying parallel with the chief line of extension. If a sheet is stretched uniformly in every direction a ring "fibre diagram" is obtained, whilst a twisted rubber rod gives a spiral "fibre diagram." If a piece of stretched rubber is gradually heated the interference spots decrease in intensity and disappear just when the tension disappears and the rubber becomes plastic; cooling to $-4°$, however, does not affect the intensity of the interference although the tension again disappears. Mechanical working, *i.e.*, mastication, of rubber causes a gradual decrease in the intensity of the interference of the stretched material until finally it is no longer observable.

D. F. Twiss.

XV.—LEATHER; GLUE.

Fluorescence test for natural vegetable tanning extracts in ultra-violet light. O. Gerngross and G. Sándor (Collegium, 1926, 1—10; cf. Gerngross, Bán, and Sándor, B., 1926, 23).—A large number of natural tanning extracts show fluorescence. Not only quebracho and tizerah but also pine bark, malet bark, and donga extract in neutral solution show slight but distinct fluorescence in ultra-violet light. In slightly alkaline solution, the bluish fluorescence of pine bark and malet bark is increased appreciably and is changed to a bright green, whilst the fluorescence of donga extract changes to a beautiful orange-red. The yellow fluorescence of fibrous tissues treated with quebracho or tizerah solutions is not confined to these two materials, but is also exhibited by fibres treated with mimosa, chestnut, and hemlock extracts but not in so marked a degree. Cotton wool dipped in pine bark and malet bark extracts shows a beautiful violet fluorescence. This is also given to a smaller extent by oakwood extract. On mixing yellow and violet fluorescing extracts (*e.g.*, equal volumes of pine bark and quebracho extracts) and treating wadding with the mixture, a snow-white fluorescence is produced. Mangrove bark extract gives a slight whitish fluorescence. The violet fluorescing substance will combine only loosely with hide powder so that it can be separated from tannin by detannisation. The fluorescent material is fixed irreversibly by cotton wool. Sulphite-cellulose extracts in aqueous solution exhibit a change in colour from blue to green on adding alkali in the same way as pine bark and malet bark, but the latter can be differentiated from sulphite-cellulose because cotton wool treated with sulphite-cellulose extract gives scarcely any fluorescence and the slight fluorescence produced can be removed by washing. D. Woodroffe.

Comparative durability of chrome- and vegetable-tanned sole leathers. R. C. Bowker and M. N. V. Geib (U.S. Bur. Standards Tech. Papers, 1925, 19, [286], 267—286).—Hides were prepared for tanning in the usual way, cut down the back, one side of each was tanned with a blend of hemlock, chestnut, wattle bark, and quebracho; the other side was chrome-tanned. Some of the chrome-tanned sides were filled with greases and mineral fillers. Under the test conditions, chrome-tanned leather, regardless of filling, wore approximately twice as long as vegetable-tanned leather per unit of thickness. Natural chrome-tanned leather gave the longest wear ratio, which decreased according to the types of filling materials used. Chrome-tanned leather filled with 25% of paraffin wax wore 115% longer than vegetable-tanned sole leather, whereas chrome leather filled with grease, glucose, and barium sulphate only showed 23% longer wear. The chrome leather had a content of hide substance double that of the vegetable-tanned leather. The chrome leather was more hygroscopic. Fillers prevented the rapid absorption of water. Natural chrome leathers swelled in water 25%, whilst filled leathers showed only 11% swelling. Natural chrome leathers in wear became very soft and pliable, had a tendency to spread, fray, and curl up at the edges, allowed water to penetrate rapidly, and wore slippery on wet surfaces. Paraffin wax-filled chrome leather was similar but waterproof. It lost this property during wear. Leather filled with paraffin wax and pyroxylin retained its water-resisting property longer. The average thickness of vegetable-tanned bends was 25% greater than that of the chrome-tanned. Topographical charts have been made to represent the variation of the thickness of the leather in sides from the same hide but of different tannages. The filling materials increase the thickness of the leather. Very heavy hides are necessary to produce chrome leather $\frac{9}{48}$in. to $\frac{11}{48}$in. thick. A hide tanned by the chrome process will yield from 10—12% less area in leather than if tanned by the vegetable process.

D. Woodroffe.

Analytical standards for leather manufacture. A. M. Goldenberg (Collegium, 1926, 10—21).—The following standards for sole leather and Russia leather have been adopted for introduction into the factories controlled by the Ukraine Leather Trust. *Soaks*: g. per litre, proteins 0·5, ammonia 0·03, sulphides (as Na_2S) 0·3. *Lime liquors (first lime)*: g. per litre, proteins 5 (sole leather), 7 (Russia leather), ammonia 0·1, lime 4, total alkalinity 3·5. *Sole leather pelts*: hide substance 28%, lime and ash <1%, total alkalinity (as NaOH) 0·8%. *Russia leather after colouring liquors*: degree of tannage 15%, hide substance (for 18% water) 70%, lime <0·3%, water-soluble matter 2—2·5%. *Sole leather after colouring liquors*: degree of tannage 25—30%, hide substance 55%, lime <0·3%, water-soluble matter 4%. *Hide after layers*: degree of tannage 55—60%, hide substance 45%, water-soluble matter 6%. *Colouring liquors (sole leather)*: *d* 1·007—1·023, tannin 0·15—2·5%, acid (as acetic) 0·5%. *Colouring*

liquors (*Russia leather*): *d* 1·004—1·011, tannin 0·10—0·15%, acid (as acetic) 0·0—0·05%. *Pit liquors, first layer*: *d* 1·021—1·028, tannin 2·5—3·0%, acid 0·5%; *second layer*: *d* 1·028—1·035, tannin 3·5—4·0%, acid 0·3%. *Drum liquors for sole leather*: *d* 1·084—1·098, tannin 13%. *Sole leather*: water 18%, ash 1·5%, fat 1·2%, water-soluble matter 14—17%, leather substance 65·3%, degree of tannage 85—90%, "rendement" figure 290%, hide substance 35·5—34·4%, combined tannin 29·8—30·9%, water resistance 30%. *Russia leather*: water 18%, ash 0·8%, fat 14%, water-soluble matter 8%, leather substance 59·2%, degree of tannage 60—66%, "rendement" figure 270—278%, hide substance 37·1—35·9%, combined tannin 22·2—23·3%.

D. Woodroffe.

Fumaric acid as a hydrolytic product of gelatin. Abderhalden and Haas.—See A., Mar., 312.

Patents.

Treatment of chrome leather waste for use in the manufacture of glue. E. Meier (Swiss P. 112,400, 8.8.24).—Waste from chrome leather which has been treated with formaldehyde is prepared for glue manufacture by treatment successively or simultaneously with alkali solution and lime.

L. A. Coles.

Process for treating blood. A. Sternberg (E.P. 224,227, 27.10.24. Conv., 29.10.23).—Coagulated blood, preferably defibrinised, is brought in the wet condition (50% of water being the most favourable proportion) to the highest degree of fineness by triturating it with wire brushes, and is then dried. A suitable device consists of two intermeshing brushes rotating in opposite directions, and preferably at different speeds. The product is specially suitable for making artificial horn etc.

D. G. Hewer.

Utilising casein- and other protein-formaldehyde compounds. Internat. Galalith-Ges. Hoff & Co., Assees. of A. Bartels and G. Eberhardt (G.P. 419,536, 23.10.23).—Protein-formaldehyde compounds, *e.g.*, artificial horn scrap, are treated with substances, such as bisulphites, which have a greater affinity than proteins for formaldehyde, and of which the formaldehyde compounds do not harden proteins. The protein-formaldehyde compound may first be treated with a protein solvent, *e.g.*, an acid or alkali. The recovered protein may be used again for preparing protein masses to be hardened with formaldehyde.

A. Geake.

Dyeing leather (G.P. 422,465).—See VI.

XVI.—AGRICULTURE.

Plate counts of soil micro-organisms. N. R. Smith and S. Worden (J. Agric. Res., 1925, 31, 501—517).—The use of an electric vibrator for pre-preparing suspensions of soil for making counts of micro-organisms (cf. Whittles, B., 1923, 466 a) is unsatisfactory on account of the difficulty of ensuring complete sterilisation of the apparatus. When this is got over, counts from suspensions prepared in this way agree quite closely with those obtained by the usual method of hand shaking. Soil extract–agar gives higher and more uniform results than other media. Field soils of fair fertility, irrespective of their general character, are suitable for the preparation of the soil extract–agar medium. Soil samples taken horizontally from a level about 4 in. below the surface give slightly higher and more uniform counts than those taken vertically with an auger.

C. T. Gimingham.

Ammonia content of soil, and its relation to total nitrogen, nitrates, and soil reaction. H. J. Harper (J. Agric. Res., 1925, **31**, 549—553).—No correlation was observed between the ammonia content and the content of total nitrogen or nitrates or the p_H value of some 40 soils investigated. The ammonia content is evidently in equilibrium with the products of protein decomposition and nitrification. The majority of soils contain less than 10 pts. per million of ammonia and the amount very rarely exceeds 20 pts. per million. C. T. Gimingham.

Formation of humus in dead surface layers of forest soils. A. Nemec (Compt. rend., 1926, **182**, 590—592).—Surface layers of the soil in forests have been investigated by treatment with hydrogen peroxide, which renders humic substances soluble in water. The proportion of humus in the organic matter of the soil increases as the acidity of the soil decreases. The rate of nitrification follows the same general rule. The p_H of the soils investigated ranged from 3·8 to 6·5, the humus content from 20·7 to 82·2% of the organic matter in the soil, and rate of nitrification from —10·3 to +194 mg. of nitrogen gained in 30 days in 1 kg. of soil (calculated on the dry weight). L. F. Hewitt.

Decomposing action of peat on phosphorite. D. Prianischnikov (Fortschr. Landw., 1926, **1**, 1—4; Chem. Zentr., 1926, I., 1612).—When calcium sulphate is added to peat, the hydrogen ions of the humic acid present are replaced by calcium and a more acid reaction is developed; in the presence of phosphorite, water-soluble phosphorus is formed. If the amount of calcium sulphate is adjusted so as not to exceed the capacity of the peat to remove calcium ions from solution, the phosphorite can be completely decomposed. Peat in presence of phosphorite and calcium sulphate becomes more acid and yields 30% more water-soluble phosphorus than with phosphorite and potassium chloride. These results were confirmed by vegetation experiments.

C. T. Gimingham.

Industrial wastes as manures. I. Ajowan and mohua cakes as fertilisers. II. Utilisation of refuse. R. D. Rege (J. Ind. Inst. Sci., 1925, **8A**, 245—265).—Ajowan cake, from the seeds of *Carum copticum*, contains about 0·9% of nitrogen, which is in a readily available condition. Traces of thymol are present, but the amount is insufficient to check bacterial activity in the soil. Mohua cake,

from the seeds of *Bassia longifolia*, contains 2·7% of nitrogen and about 8·0% of oil. When mixed with activated sewage sludge and aerated for 6 days, no decomposition of the protein of the cake occurs. The same result is obtained if the oil is first extracted. The non-availability of the nitrogen was traced to the presence of a saponin which inhibits bacterial action. Experiments with town refuse (in India), containing from 0·8 to 1·7% of nitrogen and small quantities of phosphoric acid and potassium, indicate that about 75% of the nitrogen is in a comparatively unavailable condition. In mixtures of powdered refuse and activated sewage sludge to which ammonium sulphate was added, nitrification took place rapidly and the soluble nitrogen was removed from solution. There was no loss of nitrogen if the proportion of carbon was not too high. It is suggested that raw sewage might be treated with sludge and refuse material with the object of conserving the soluble nitrogen. C. T. Gimingham.

Method of obtaining size distribution of particles in soils and precipitates. D. Werner (Trans. Faraday Soc., 1925, 21, 381—394).—See B., 1926, 31.

Patents.

Manufacture of phosphatic fertilisers. Chem. Fabr. Heppes u. Co., and J. B. Carpzow (G.P. 421,271, 7.7.23).—Fertilisers are produced by drying and grinding mixtures of phosphates with fresh sludge containing a high percentage of colloidal silica, such as sapropel or saprocol. L. A. Coles.

Manufacture of a stable mixed fertiliser containing calcium nitrate. Badische Anilin- & Soda-Fabr., Assees. of H. Weitzel (G.P. 421,331, 31.7.24).—Calcium nitrate is mixed with about two equivalents of ammonium phosphate or ammonium sulphate, or mixtures of these, to form non-deliquescent fertilisers. L. A. Coles.

Manufacture of fertiliser containing urea. A.-G. für Stickstoffdünger (G.P. 421,852, 27.1.22).—The fertiliser is produced by treating calcium cyanamide with acids and water or steam, at a temperature and under conditions such that water not combined, or liberated during the process, is removed by evaporation. L. A. Coles.

Attractant for insects. L. B. Smith, E. A. Richmond, and P. A. van der Meulen, Assrs. to the People of the United States (U.S.P. 1,572,568, 9.2.26. Appl., 12.8.25).—A preparation containing geraniol as the active ingredient is claimed as an attractant for insects, particularly for the Japanese beetle. C. T. Gimingham.

Utilisation of end liquors from crude potassium salts (G.P. 422,987).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Influence of salts on the crystallisation of sugar. W. J. Kusnetzov (Z. Ver. Deuts. Zucker-Ind., 1926, 19—24).—Alkali carbonates inhibit the crystallisation of sucrose from its aqueous solution, sodium carbonate to a greater degree than the potassium salt, this effect diminishing with falling temperature. Calcium nitrate and aspartate behave similarly. On the other hand, the presence of calcium chloride promotes crystallisation, though to a comparatively small extent. These effects are influenced by the viscosity of the solution. J. P. Ogilvie.

Treatment of molasses with zeolites in applying the Steffens process. F. R. Bachler (Ind. Eng. Chem., 1926, 18, 180—182).—Treatment of beet molasses with zeolites, so as to remove the whole or greater part of the alkali salts, previous to submitting it to the Steffens desaccharification process, is advocated. J. P. Ogilvie.

Commercial caramels and molasses colouring substances. H. Simmich (Z. Ver. Deuts. Zucker-Ind., 1926, 1—18).—German commercial caramels were found to contain caramelan, caramelen, and caramelin (cf. Cunningham and Dorée, B., 1917, 973); some nitrogenous substances were also present, probably originating from the ammonia used in the manufacture of the caramel. Absorption curves determined with the König–Martens spectrophotometer did not show resemblance to those of "Kassler Braun," a humic acid prepared from lignite. Beet molasses colouring matters were precipitated with lead acetate, and three colouring substances containing nitrogen were isolated; these gave the characteristic absorption curves of the colouring matters found in commercial caramels. A fourth substance separated showed absorption phenomena similar to those observed with humic substances. J. P. Ogilvie.

Studies on starch. C. L. Alsberg (Ind. Eng. Chem., 1926, 18, 190—193).—Most varieties of starch when heated in water suspension do not disintegrate to form a colloidal solution, the granules merely swelling gradually (cf. Harrison, B., 1911, 534). If natural starch be ground in a pebble mill until most of its granules are injured, though still recognisable under the microscope as starch, it becomes incapable of yielding paste in ordinary concentration. Most of the starch granule substance has become colloidally soluble in cold water. From such ground starch without heat or the use of any reagent clear solutions can be obtained in cold water, these containing material which gives the characteristic iodine reaction, does not reduce Fehling's solution, and is capable of dialysing through thin colloidal membrane. J. P. Ogilvie.

See also A., Mar., 273, **Revision of structural formula of dextrose** (Charlton, Haworth, and Peat). 275, **Relationship between optical rotatory power and structure in chemistry of polysaccharides** (Pringsheim and Leibowitz). 276, **Amylose octadextrin** (Klason and Sjöberg).

Patents.

Refining of sugar. A. W. Mathys. From F. Kessler (E.P. 246,976, 23.12.24).—Raw sugar is subjected to a sudden change in temperature, for

example, by treatment with a large volume of superheated steam, whereby innumerable fine fissures are produced in the crystals, or they are broken into fragments. The crystals are then readily purified by treatment with a pure sugar solution or one containing refining agents. If the refining agents be added to the raw sugar the pure sugar solution may be produced *in situ* by the action of the steam. D. G. HEWER.

Manufacture of cane-sugar [from molasses]. H. S. PAINE, C. F. WALTON, JUN., and V. BIRCKNER; dedicated to Citizens OF U.S.A. (U.S.P. 1,572,359, 9.2.26. Appl., 8.2.24).—The invert sugar present in molasses is destroyed by selective fermentation and the sucrose is then precipitated as saccharate, which is worked in the usual way. A. R. POWELL.

Producing sugar from cellulose. N. KRANTZ and L. DE MOLTKE-HUITFELDT (F.P. 595,439, 10.3.25).—Cellulose-containing substances, *e.g.*, sawdust, are treated with dry hydrogen chloride in the presence of water or the like. A. GEAKE.

Treating sugar juices or the like with hyposulphite. R. DUTILLOY (F.P. 595,714, 30.6.24).—Crude diffusion or press juices, or syrup or mother liquors from crystallisation, are treated while neutral or feebly acid, with a determined amount of hyposulphite at 50—60°. A. GEAKE.

Treating sugar-containing juices or solutions to decolorise or purify them, or render them suitable for filtration. C. W. SCHONEBAUM (Dutch P. 12,990, 9.11.21).—The juice or solution is treated with ozone at a raised temperature in the presence of a substance which will combine with acids and keep the solution neutral. Decomposition of sugar is thereby avoided. A. GEAKE.

XVIII.—FERMENTATION INDUSTRIES.

See A., Mar., 324, **Relation between alcoholic fermentation and hydrogen-ion concentration** (HÄGGLUND and AUGUSTSON); **Formation of acetylmethylcarbinol and βγ-butylene glycol** (LEBEDEV); **Synthesis of coproporphyrin by yeast** (FISCHER and FINK); **Biogens, inactive mother-substances of the two "bios"** (EASTCOTT); **Reproduction of yeast in solutions to which no bios had been added** (WHITEMAN); **Influence of electrolytes on electrophoretic migration of bacteria and of yeast cells** (WINSLOW and FLEESON); **Acetic fermentation** (NEUBERG and WINDISCH). 325, **Mechanism of lactic acid formation by bacteria** (NEUBERG and GORR); **Fermentation of cellulose by thermophilic bacteria** (VILJOEN, FRED, and PETERSON). 328, **Viscosimetric determination of amylase** (DAVISON).

PATENTS.

Preparing yeast nutrient solutions and manufacturing yeast therefrom. R. L. CORBY and R. GLASGOW, Assrs. to FLEISCHMANN Co. (U.S.P. 1,571,932, 9.2.26. Appl., 13.3.19).—To a nutrient solution consisting of molasses, cereal material, and nutrient salts, and having an acidity higher than the optimum for yeast propagation, a compound containing inorganic yeast-assimilable nitrogen and having an alkaline reaction is added, in amount requisite to produce the most satisfactory conditions for yeast propagation in respect to acidity and content of assimilable nitrogen. D. G. HEWER.

Denatured alcohol. H. C. FULLER (U.S.P. 1,573,697, 16.2.26. Appl., 6.6.21).—The denaturant is a halogen-containing glyceryl ester. H. HOLMES.

Denatured alcohol. H. C. FULLER, Assr. to UNION CARBIDE AND CARBON RESEARCH LABORATORIES (U.S.P. 1,573,698, 16.2.26. Appl., 21.6.22).—The denaturant is a halogen fatty acid ester of a polyhydric alcohol. H. HOLMES.

Manufacture of yeast. C. HOFFMAN, N. M. CREGOR, and H. D. GRIGSBY, Assrs. to FLEISCHMANN Co. (U.S.P. 1,575,761, 9.3.26. Appl., 22.3.22).—See E.P. 195,347; B., 1923, 1039 A.

XIX.—FOODS.

Nature of the agglutination of fat globules. IV. Relation of serum globulins to the "creaming" of milk. E. BROUWER. **V. Influence of natural and separated skim milk on the creaming of washed milk-fat globules.** E. HEKMA (Jaarversl. 1924, Vereen. tot. exploit. proef. te Hoorn, 1925, 18—35; 36—43; Chem. Zentr., 1926, I., 524).—The two proteins, euglobulin and pseudoglobulin (prepared from the blood serum of cattle), when added to milk, differ markedly in their effect on the rising of the cream. The former greatly assists the process, whereas the latter has little action. The blood serum of new-born calves, which contains scarcely any euglobulin, has little or no effect. There is not, however, a direct correlation between the amount of euglobulin in blood serum and the power of promoting "creaming." The possibility that euglobulin plays a part in the natural "creaming" of milk is discussed.

Skim milk from a separator has a greater influence on the agglutination of washed milk-fat globules than natural skim milk and this difference is still noticed if the fat globules are heated to 80—90° for 5 min., thus causing a considerable reduction in the capacity for "creaming." It appears that some substance derived from the leucocytes in the milk is concerned with "creaming." C. T. GIMINGHAM.

Leucocyte content and catalase number of natural and separated cream. E. HEKMA (Jaarversl. 1924, Vereen. tot. exploit. proef. te Hoorn, 1925, 4—10; Chem. Zentr., 1926, I., 524).—The number of leucocytes in natural cream is very much greater than in separated cream—approximately 4,000,000 per c.c. in the former and 35,000 in the latter. The catalase number is also much

higher in natural cream. The conditions are reversed in the corresponding skim milks.
C. T. GIMINGHAM.

Chloropicrin as a fumigant for cereal products. R. N. CHAPMAN and A. H. JOHNSON (J. Agric. Res., 1925, 31, 745—760).—The high toxicity of chloropicrin to the granary weevil is demonstrated. A lethal concentration can be rapidly obtained, even in large bulks of cereal products, if the chloropicrin is atomised. The presence of very small amounts in flour affects the quality of the bread; not only is the physical condition of the gluten changed but yeast activity in the dough is retarded. Disappearance of chloropicrin from fumigated flour and wheat is slow but eventually complete, given proper exposure to the air. C. T. GIMINGHAM.

Apparatus for the determination of water [in foodstuffs, fat, etc.]. H. KREIS (Ann. Chim. Analyt., 1926, 8, 33—34).—An apparatus for the determination of water by distillation with toluene or similar immiscible liquid, consists of a flask carrying an upright tube of large diameter with a side tube extending from a point just above the cork of the flask to a point above the open upper end of a smaller graduated tube within the wide tube. The upper end of the wide tube carries a condenser. The arrangement is such that the vapours pass only up the side tube, are condensed, and the condensed liquid collects in the graduated inner tube. D. G. HEWER.

Rapid determination of water content [in cereals etc.]. J. TAUSZ and H. RUMM (Z. angew. Chem., 1926, 39, 155—156).—The substance is mixed with dry tetrachloroethane in a flask carrying a 3-bulb fractionating column, the upper end of which is bent over and elongated to form the inner tube of a Liebig condenser. The distillate is collected in a tube with levelling arm, and the supernatant water finally forced up into a narrow graduated tube and measured. The method is particularly useful in the case of cereals and other substances which give up their moisture with difficulty in an oven at 105°. W. T. K. BRAUNHOLTZ.

Testing and estimation of decay in preserved eggs. A. SCHMID (Mitt. Lebensm. Hyg., 1925, 16, 137—143; Chem. Zentr., 1926, I., 1320).—Satisfactory results are obtained by the following method, due to Sudenorf (private communication): 1—2 g. of the dry substance are warmed with 30—60 c.c. of neutral ether-alcohol, and titrated quickly with 0·1*N*-sodium hydroxide solution in the presence of phenolphthalein, till a strong red colour persisting for 30 sec. results. The acidity thus determined is a measure of the deterioration. J. GRANT.

Determination of unsaponifiable matter in flour, alimentary pastes, and eggs. R. HERTWIG and L. H. BAILEY (J. Assoc. Off. Agric. Chem., 1926, 9, 122—124).—The direct ether extract of wheat flour and eggs does not contain all the unsaponifiable matter of these materials, and the acid hydrolysis method for extraction of fat yields low results for flour and eggs. The method of Rask and Phelps (B., 1925, 224) and Hertwig's "neutral" method (B., 1923, 1040 A) yield quite similar results. The A.O.A.C. neutral method for the lipoid extraction of flour, alimentary paste, and eggs is shown to be the most satisfactory. The lipoids from 5 g. of wheat flour or alimentary paste, or from 10 g. of liquid eggs or 2 g. of powdered dried egg, are extracted according to the A.O.A.C. methods (1925, 40, 58), and the unsaponifiable matter is determined either on the dried crude extract or on the purified and weighed lipoids (cf. B., 1925, 600). C. O. HARVEY.

Effect of chemical preservation of eggs upon the stability of their vitamin contents. E. TSO (Biochem. J., 1926, 20, 17—22).—In the Chinese preserved egg "pidan" (ducks' eggs preserved in a mixture of slaked lime, straw ash, salt, and water), vitamin-B is practically completely destroyed whilst vitamin-A and vitamin-D are hardly affected.
S. S. ZILVA.

Variations in individual sugars in the Jerusalem artichoke during growth. S. H. COLLINS and R. GILL (J.S.C.I., 1926, 45, 63—65 T).—Following investigations on oat straw (J.S.C.I., 1922, 56 T) artichokes have been grown and analysed. The tubers contain much inulin, and the stalks much lævulose at active stages of growth, but mere traces when growth is over and the tubers are formed. The relative proportions of dextrose and lævulose in artichokes are reversed to those in oats. Detailed figures are given, showing the composition of both stalk and tubers at all stages of growth. A striking difference from cereal grains, or potatoes, is the continued increase in percentage of proteins in the tubers at late stages of development.

Occurrence of arsenic in apples. H. E. COX (Analyst, 1926, 51, 131—137).—Out of 39 samples of apples (American) only 5 were found free from arsenic, which was present in proportions ranging from 0·5 pt. to 15 pts. As_2O_3 per million. The proportions of lead found in the samples examined indicated that the fruit had been sprayed with lead arsenate. The distribution of arsenic on apples which had been heavily sprayed with lead arsenate in the laboratory, and then suspended for 48 hours, was in two cases: whole apple, 24 and 7 pts. per million; on the skin 100 and 33, and in the "flesh" 3·3 and 1·3. In spite of washing sprayed apples in running water a small but definite retention of arsenic was found, but if 2% caustic soda solution is used for washing, the removal of the arsenic is almost complete.
D. G. HEWER.

Examination of cacao. H. LÜHRIG (Pharm. Zentr., 1926, 67, 129—135).—Many samples of cacao beans and cocoa from industrial and other sources have been examined chemically and microscopically. Samples of material taken at different stages during treatment in a Bauermeister refining machine showed that the husk-content of the broken and purified material increased with increased fineness of grinding. Tables are given of the analyses and microscopical examination of various samples,

including a very extensive table of the iron (ferric oxide), manganese (manganous oxide), and sand content of many samples (nibs, husks, and cocoa).

B. Fullman.

Vitamin content of plant juices. R. Sucharipa (Konserven-Ind., 1925, 12, 623—624; Chem. Zentr., 1926, I., 1594).—Vitamins are not more stable in fruit juices prepared without heating than in cooked juices, on account of the action of oxydases in the former. For preservation of the vitamin content, the fruit should be rapidly pressed and the juice collected in small closed containers. Sterilisation should be carried out immediately. The concentration of sugar in the final product should not exceed 35% and storage should be in a cool, dark place.

C. T. Gimingham.

Formaldehyde in marine products. D. B. Dill and P. B. Clark (J. Assoc. Off. Agric. Chem., 1926, 9, 117—122).—The natural development of formaldehyde in canned *Crustacea*, discovered by Ishida (J. Pharm. Soc. Japan, 1917, [422], 300), has been confirmed by the authors, who show that this development is not related to the growth of organisms, and that it is not affected by the nature of the container. It takes place in the absence of free oxygen. Concentrations of formaldehyde in canned *Crustacea* as high as 1 pt. in 30,000 have been found, and, as acidification and steam distillation failed to recover more than one-third of the formaldehyde added to formaldehyde-free salmon, it is concluded that the concentration of formaldehyde actually present may be as high as 1 pt. in 10,000. Formaldehyde formation has also been found to take place in canned red rock cod (*Sebastodes* sp.) and herring, but not in sardine, salmon, and other canned fish meat.

C. O. Harvey.

See also A., Mar., 313, **Van Slyke's method of determination of nitrogen distribution in proteins; Distribution of nitrogen in proteins of eggs; Direct determination of arginine** (Plimmer and Rosedale). 321, **Milk-diastase** (Chrzaszcz and Goralowna). 326, **Accessory growth factors** (Von Euler and Steffenburg); **Vitamin-B and metabolism** (Plimmer and Rosedale).

Patents.

Flour bleaching gas. F. Stacey (E.P. 246,979, 5.1.25).—Nitrogen peroxide is generated electrically in a tube with a contracted passage. The electrodes are placed within the tube in front of the contraction and a nozzle fed from an air compressor is placed between them. The electrodes are supplied with electrical energy by a magneto.

D. G. Hewer.

Preservation of fresh fruit and vegetable. H. R. Fulton and J. J. Bowman (U.S.P. 1,571,938, 9.2.26. Appl., 3.10.25).—An aqueous solution containing the sodium oxide and boric acid radicals in equal proportions and of such strength as to retard the development of organisms causing stem and blue mould rots and other forms of decay (*e.g.*, 5 pts. by weight of borax and 1·39 of anhydrous sodium carbonate in 100 of water) is applied to the surface of fresh fruits and vegetables.

D. G. Hewer.

Pectin. Distillerie des Deux-Sèvres (F.P. 595,349, 11.6.24).—Potatoes or carrots are boiled with acidified water, and the mass is filtered and pressed. The extract is evaporated first to *d* 1·050—1·060, and then to *d* 1·300.

A. Geake.

Recovering oil or fat from milk and cream. A. E. White. From Milk Oil Corp. (E.P. 247,617 and 247,660, 30.11.23).—See U.S.P. 1,485,701 and 1,494,698; B., 1924, 397, 690.

Treating blood (E.P. 224,227).—See XV.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Valuation of digitalis. R. Wasicky, F. Lasch, and K. Schonovski (Arch. Pharm., 1926, **264**, 92—98).—Chiefly a review in which chemical and biological methods are compared. The colorimetric method of Knudson and Dresbach (*cf.* A., 1922, ii., 882), which depends on a colour reaction with picric acid in alkaline solution, is shown to be valueless for testing the crude drug, though it is of some value when pure glucosides are under investigation. For commercial digitalis preparations only biological methods give satisfactory results, and the method of Pick and Wasicky (Wiener Med. Woch., 1917, 6) is recommended because of its comparative simplicity and the rapidity with which it is carried out. There is a definite connexion between the bitterness of a digitalis extract and its potency as a medicament. The value of this as the basis for a simple test is being investigated.

W. A. Silvester.

Distillation of aqueous formaldehyde solutions. E. W. Blair and R. Taylor (J.S.C.I., 1926, 45, 65—66 T).—Aqueous formaldehyde solutions below 30% strength (by vol.) yield distillates of higher concentration than the residual liquors, whilst the reverse holds for solutions above 30% strength. The 30% formaldehyde solution approximates to a mixture of constant minimum boiling point (about 98·7°).

Essential oil of *Daucus carota*, L. I. Y. Asahina and T. Tsukamoto (J. Pharm. Soc. Japan, 1925, [525], 1—4; Chem. Zentr., 1926, I., 1820).—Oils from the fruit, fruit and stalks, and stalks alone have d^{22}, 0·9088, 0·9270, 0·9584; $[\alpha]_D^{22}$—5·98°, +1·04°, +5·06°; acid values nil, 2·86, 24·91, and saponif. values 74·08, 69·69, 65·97, respectively. An oil from the fruit and stalks (d^{22} 0·9220, acid value 1·17, saponif. value 66·0, acetyl value 156·8) on fractionation under 7 mm. pressure gave six fractions between 110° and 148°. Each fraction on oxidation with potassium permanganate gave a trihydric sesquiterpene alcohol, $C_{15}H_{28}O_3$. The corresponding monohydric alcohol was isolated from the oil as its mercuric acetate compound, from which the

hydroxide, R·Hg·OH, was obtained by treatment with alkali. The hydroxide, after purification through the chloride, R·HgCl (m.p. 194°), was a starch-like powder m.p. 203° (sulphate, m.p. 135—140°; nitrate, m.p. 153—155°), which on treatment with alcoholic hydrogen sulphide yielded the sesquiterpene alcohol *carotol*, $C_{15}H_{26}O$, b.p. 109°/1·5 mm., d_4^{15} 0·9646, n_D^{15} 1·4912, $[\alpha]_D+27·9°,+28·03°$ (1·491 g. in 10 c.c. of alcohol, l = 0·5 dm.). Judging from the molecular refractivity and non-absorption of hydrogen (H + Pt) it appeared to be saturated, but potassium permanganate oxidised it to the above trihydric alcohol *dihydroxycarotol*, $C_{15}H_{28}O_3$, m.p. 142°, $[\alpha]_D$—2·41° (*monobenzoate*, plates, m.p. 83—83·5°, $[\alpha]_D+$ 27·36°). Carotol and bromine in glacial acetic acid yielded a *dibromide* (prisms, m.p. 58·5°), which on boiling with two mols. of silver benzoate and hydrolysis of the product gave daucol, $C_{15}H_{26}O_2$, m.p. 118·5°, $[\alpha]_D$—15·10° (acetyl derivative, m.p. 81·3°). Richter (B., 1909, 1222) gives 116° and 79° respectively. As daucol is very stable and carotol easily loses water the following explanation of the above reactions is suggested:

$$\underset{\text{Carotol.}}{R{\cdot}OH} \rightarrow \underset{\text{Dihydroxycarotol.}}{R(OH)_3} \rightarrow \underset{\text{Daucol.}}{R(OH):O}$$

E. H. SHARPLES.

Oil from leaves and twigs of the lemon-tree (petitgrain). P. LIOTTA (Riv. Ital. essenze e profumi, 1925, 7, 107—108; Chem. Zentr., 1926, I, 1309).—The oil prepared by the author had d^{15} 0·8668, and α^{20} 24°15′, acid value 0·97, as acetic acid 0·11%, ester value 51·7, as linalyl acetate 18·10%, citral (Romeo method) 13·27%, and was soluble in 19 vols. of 80% alcohol. E. H. SHARPLES.

Essential oils of the Crimea. B. RUTOVSKI, I. WINOGRADOVA, and A. KONDRATSKI (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 59—86; Chem. Zentr., 1926, I, 1304—1306).—A survey of the Crimean essential oils and observations on the influence of previous comminution of the plants on the yield of oil by distillation. E. H. SHARPLES.

Essential oils from Sukhum district [Caucasia]. B. RUTOVSKI, I. WINOGRADOVA, and W. KOSLOV (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 93—117; Chem. Zentr., 1926, I, 1306—1307).—Physical and chemical characteristics of 23 essential oils are given. E. H. SHARPLES.

Essential oils of Sochi district [Caucasia]. B. RUTOVSKI, I. WINOGRADOVA, and G. KOLOTOV (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 118—126; Chem. Zentr., 1926, I, 1307—1308).—Physical and chemical characteristics of 17 essential oils are given. E. H. SHARPLES.

Essential oil of *Andropogon citratus* D.C. from Sukhum [Caucasia]. W. KRASTELEVSKI (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 159—161; Chem. Zentr., 1926, I, 1308).—A yield of 0·19—0·35% of lemon-grass oil was obtained from *Andropogon citratus*, D.C. E. H. SHARPLES.

Yield of some essential oils from Sukhum [Caucasia]. W. KRASTELEVSKI (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 163—166; Chem. Zentr., 1926, I, 1308).—The following yields of oil are recorded: *Mentha pulegium*, L., 0·63% (d^{15} 0·930, $\alpha_D+12·3°$) from the fresh plant, *Mentha crispa*, L., 0·84% (d^{15} 0·8843) from the fresh plant, *Mentha piperita*, L., 2·49% from the dried plant, *Liquidambar orientalis*, 0·26% from the green fruits, *Satureja hortensis*, 0·37% from the dried plants, *Thymus vulgaris*, L., 0·28—0·36% from the dried plants, *Valeriana officinalis*, 0·36% from the roots. E. H. SHARPLES.

See also A., Mar., 305, **Action of mercuric salts on dialkylbarbituric acids** (FLEURY). 311, **Organic bismuth compounds** (MASCHMANN). 320, **Narcotic action of propylene** (HALSEY, REYNOLDS, and PROUT); **Bacterial chemotherapy, with reference to mercury dyes** (RAIZISS, SEVERAC, and MOETSCH); **Chemotherapy of bacterial infections** (WALKER and SWEENEY). 322, **Preparation of standards for colorimetric determination of pepsin** (SMORODINCEV and ADOVA). 325, **Tuberculin** (LONG and SEIBERT). 328, **Detection of bismuth in organic material** (DANCKWORTT and PFAU); **Toxicological investigation of mercury** (KOHN-ABREST); **Pharmacological determination of ergotamine content of commercial ergot preparations** (MAHN and REINERT); **Pharmacological determination of ergotamine-ergotoxin titer of ergot** (MASUDA).

PATENTS.

Manufacture of organic mercury compounds. FARBENFABR. VORM. F. BAYER & CO. (E.P. 242,669, 9.11.25. Conv., 10.11.24; cf. G.P. 234,851).—Halogen-, nitro-, or halogen-nitro-substituted phenols may be converted almost quantitatively into the corresponding nuclear-substituted mercury derivatives by running a mercury salt solution into a heated solution of the phenol in so much sodium carbonate that the reaction-mixture becomes acid only when decomposition is complete. For example, 50 parts of mercuric sulphate in about 27 pts. of sulphuric acid of d 1·84 and 140 pts. of water are allowed to flow with stirring into a solution of 24·8 pts. of *o*-nitrophenol in 47 pts. of sodium carbonate and 170 pts. of water at 75°, during 2½ hrs., maintaining the temperature at 72—75°. After stirring for 1 hr. at 70° a 96·5% yield of mercury *o*-nitrophenol (containing 55—55·5% Hg) is obtained. B. FULLMAN.

Manufacture of tetraglucosan. J. KERB (E.P. 243,348, 14.11.25. Conv., 20.11.24).—Tetraglucosan is obtained by heating dextrose in the presence of a small quantity of metal or metals or their salts other than platinum or zinc and their salts, under diminished pressure or in an inert atmosphere, preferably with an inert diluent. If iron salts be used the iron is converted into a partially insoluble compound. For example, 1 kg. of anhydrous dextrose is treated with 10 drops of 10% ferric sulphate solution and the mass mixed after keeping for a short time. 1 kg. of a mineral jelly oil is added, and the mixture heated under 15 mm. pressure, with stirring, for 1 hr. at

135°. The tetraglucosan (equivalent to 90% of the dextrose) is separated and powdered. B. Fullman.

Manufacture of a remedy for foot-and-mouth disease. A. Van der Wal (E.P. 247,018, 28.2.25).—An aqueous solution of lead monoxide and lead acetate is treated with potash alum and filtered; an excess of lead must be used, which will remain as a soluble compound. For example, a filtered solution containing 10 g. of lead monoxide and 26 g. of lead acetate per 100 g., is treated with a solution of 15 g. of potash alum, filtered, and the filtrate diluted to 1000 g. To this may be added boric acid as disinfectant; colouring matter free from tannin (such as solution of caramel or extract of chicory); substances to act as accessories and to repel insects, *e.g.*, a mixture of tinctures of eucalyptus, myrrh, and aloe, alcohol, citronella and eucalyptus oils. The above mixtures are suitable for application to the mouth. For application to the hoofs, they are thickened by, *e.g.*, the addition of a mass containing wood-tar, resin, oil (rape, linseed, etc.), beeswax, yolk (grease), zinc oxide, boric (or benzoic or salicylic) acid, eucalyptus and/or citronella oil. B. Fullman.

Production of acridine derivatives and their solutions. C. Schnorf and F. Hefti (E.P. 247,250, 14.10.24).—Derivatives of 9-aminoacridine react with carbohydrates or mixtures of the latter, with elimination of water, forming compounds soluble in water, and stable to light, air, and in solution, and possessing other therapeutic advantages over such substances as 6 : 9-diamino-2-ethoxy-acridine. For example, 289 pts. of 6 : 9-diamino-2-ethoxy-acridine hydrochloride and 180 pts. of galactose in 5000 pts. of 90% alcohol are heated on the water-bath till precipitation of the product ends. After crystallisation from methyl alcohol, and removal of the solvent by heating in air and *in vacuo*, a bright yellow *substance*, $C_{21}H_{26}O_6N_3Cl$, m.p. 208—209° is obtained. The compounds may be prepared in solution ready for use by, *e.g.*, adding 100 g. of 6 : 9-diamino-2-ethoxy-acridine hydrochloride to 400 g. of lactose dissolved in 600 g. of boiling water, boiling till solution is almost complete, filtering, and treating the cold filtrate with 75 g. of glycerin, 25 g. of *N*-hydrochloric acid, and 100 g. of water, when the liquid is ready for use. B. Fullman.

Separation of lipoids from ovaries and similar human or animal organs. O. Fellner (G.P. 420,438, 13.1.21).—The crude product obtained from the organ by extraction with alcohol, ether, and acetone is extracted with 60—75% alcohol, and the extract evaporated. For example, the product obtained by extracting dried and powdered animal placenta successively with alcohol, acetone, alcohol, and ether in the warm, and evaporation, is dissolved in 75% alcohol. Residues of cholesterols and their esters and lipoids of unknown constitution remain undissolved, and on concentration there is obtained a physiologically active mixture of lipoids as a brown, viscous, fatty mass, soluble in alcohol, ether, acetone, etc. (cf. A., 1924, i., 1017). B. Fullman.

Preparation of solutions of medicaments insoluble or difficultly soluble in water. Ges. für Chem. Ind. in Basel, and W. Minnich (G.P. 420,649, 13.9.24; Swiss P. 111,560, 21.8.24).—Using phenylacetylene as solvent, solutions may be prepared of camphor, cholesterol and other lipoids, quinine, pyrazolone derivatives, such as phenyldimethylpyrazolone, halogenated alcohols, such as trichlorobutyl alcohol, and oils such as sandalwood oil. Solutions of high concentration can be obtained, which are non-irritant, stable, and can be applied as injections or percutaneously. B. Fullman.

Rapid fixing of ethylene by means of sulphuric acid for the purpose of obtaining ethylsulphuric esters. A. A. L. J. Damiens, M. C. J. E. de Loisy, and O. J. G. Piette (U.S.P. 1,574,796, 2.3.26. Appl., 13.4.22).—See E.P. 180,988; B., 1923, 440 a.

Bismuth vanadate (G.P. 422,947).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Patents.

Non-static [photographic] film. H. J. Hoffman, Assr. to E. I. du Pont de Nemours and Co. (U.S.P. 1,570,062, 19.1.26. Appl., 12.10.21. Renewed 22.6.25).—The film is coated with a composition consisting of a conductivity-increasing substance in a solvent for the film, and then dried. For example, an electrolyte dissolved in a cellulose ester solvent may be applied to a cellulose ester film. A suitable electrolyte is ammonium nitrate applied in the form of a 2% solution in a cellulose ester solvent, sufficient in amount to fix 0·25% of the nitrate on the weight of the film. W. Clark.

Non-static photographic film. E. C. Pitman, Assr. to E. I. du Pont de Nemours and Co. (U.S.P. 1,570,076–9, 19.1.26. Appl., 16.9.20. 1,570,077 renewed 22.6.25).—(a) The film has a backing containing sodium acetate. (b) A transparent, flexible, thin, substantially neutral film has a backing of a carbohydrate ester, *e.g.*, cellulose acetate, containing glycerin. (c) The film backing contains a substance, such as saponin, capable of reducing surface tension. (d) The film backing contains starch acetate. W. Clark.

Light-sensitive photographic materials. S. E. Sheppard, Assr. to Eastman Kodak Co. (U.S.P. 1,574,943–4, 2.3.26. Appl., 6.6.24).—See E.P. 235,210–1; B., 1925, 694.

XXII.—EXPLOSIVES; MATCHES.

Patent.

Drying finely-divided materials [nitrostarch]. J. B. Bronstein, Assr. to Trojan Powder Co. (U.S.P. 1,573,673, 16 2.26. Appl., 23.10.19).—Nitrostarch is dried in long narrow tubular fabric bags by exposure to a drying atmosphere. Moisture evaporates from the surface of the bags, and the moisture content of the nitrostarch is reduced by capillarity. C. O. Harvey.

XXIII.—SANITATION; WATER PURIFICATION.

Water purification at East Liverpool, Ohio. D. H. RUPP (Fourth Annual Rep. Ohio Conference on Water Purif., Nov., 1924, 47—59).—The plant includes a grit chamber, 2 coagulation basins, 6 1-million gall. gravity rapid sand filters, and a pedestal type chlorinator. Coagulation removes 81—87% of the bacteria present in the raw (Ohio River) water; filtration 77—84% of the bacteria in the water supplied to the filters; and chlorination 94—97% of the bacteria surviving filtration. The river water contains an average of 4 p.p.m. of soluble iron, and there is probably several times that amount of soluble alumina. This explains the comparatively small amount of coagulant required, 0·7 to 1·5 grain per gal. Considerable difficulty has been experienced with regard to tastes due to pollution of supply by wastes containing phenol. Study of the conditions indicates that tastes complained of are phenol tastes and not chlorophenol tastes as a result of chlorination When the content of phenols, as determined with the Folin–Denis reagent after distillation, was greater than 0·1 p.p.m., there were invariably complaints from consumers. Taste appears to be independent of residual chlorine content, which varies inversely with the organic content, being less when the efficiency of coagulation decreases. Distinct removal of phenols and substances reacting with the Folin–Denis reagent is effected by the purification process as a whole. The phenolic substances removed are not in suspension, and it is believed that they are removed in the coagulation process. When the filter sand was cleaned to remove incrustation, the material removed yielded, per 100 g., 15 mg. of phenol on acidification with sulphuric acid and distillation. Chlorine treatment does not effect any removal of phenols, the phenol content of the filtered and the disinfected water being practically identical. In laboratory experiments the phenol content of distilled water was reduced from 1 to 0·5 p.p.m. by treatment with mangan-permutit. Phenol appears to undergo oxidation on long contact with river water; hence proximity to source of pollution is a factor in taste production. R. E. THOMPSON.

Experiences in the operation of the new municipal water softening plant at Newark, Ohio. C. T. KAISER (Fourth Annual Rep. Ohio Conference on Water Purif., Nov., 1924, 60—64).—The plant (capacity 6½ million gals.) consists of mixing chambers, Dorr clarifier, settling basins, rapid sand filters, and chlorinator. Split chemical treatment is employed, an average of 9 grains of lime, 1·5 of soda ash, and 1 of alum per gal. being applied to one-third of the raw water flow; after 20 mins. the treated portion is mixed with the remaining two-thirds of the raw water. Hardness is reduced from average of 275 to 85 p.p.m. When incrustant hardness is less than 40 p.p.m. the use of soda ash is dispensed with. An average removal of 98·5% of the suspended solids is effected by the Dorr clarifier. Carbonation is effected by means of carbon dioxide applied through diffusers in the outlet of the settling basins. The use of scrubbed flue-gas from hand-fired boilers for this purpose was found to give rise to chlorophenol tastes after chlorination, and a coke furnace has been installed for generation of carbon dioxide, 100 lb. of coke being required per million gals. The gas applied contains an average of 5% CO_2, while that emerging as bubbles from the carbonation chamber contains 0·5%. The process is controlled by hourly tests of the phenolphthalein alkalinity, a concentration of approx. 1 p.p.m. of carbon dioxide being maintained in the water applied to the filters. If free carbon dioxide exceeds 2 p.p.m., an increase in the total alkalinity of the filtered water occurs, undoubtedly due to re-solution of the "mat" on the filters. R. E. THOMPSON.

New water purification plant at Toronto, Ohio. D. H. RUPP (Fourth Annual Rep. Ohio Conference on Water Purif., Nov., 1924, 65—67).—The plant consists of mixing chamber, coagulation basins, gravity rapid sand filters, and chlorinator. Double coagulation is used at all times, this having been found to give the best results with least application of chemicals. The amount of alum used has averaged 3 grains per gal. in the approximate ratio of 2 : 1 to first and second basin, respectively; the lime used has averaged 2 grains per gal. With the exception of the first month of operation the filtered water before chlorination has met the requirement of the original U.S. Publ. Health Service standard (2 *B. coli* per 100 c.c.), and after disinfection with approximately 0·2 p.p.m. of chlorine, the final effluent has easily conformed to the revised standard (1 *B. coli* per 100 c.c.). R. E. THOMPSON.

Use of sodium aluminate as a coagulant. (1) J. P. BROWNSTEAD. (2) E. T. EDWARDS (Fourth Annual Rep. Ohio Conference on Water Purif., Nov., 1924, 31—36).—(1) During a 4-month period in 1924 when aluminate and alum were used, the cost of coagulants averaged $9 per million gals., and the reduction in bacteria was more than 90%, compared with a cost of $12 and bacterial reduction of 70% with alum and lime over a period in 1922 when somewhat similar river conditions prevailed. (2) The results of a 2-month experimental period during which aluminate was substituted for the lime ordinarily used with alum are summarised. When the coagulant was applied in the primary and secondary basins in ratio of 2 to 1 respectively, a slightly higher efficiency was obtained in the primary basin, the floc settling more rapidly and producing a clearer effluent. This resulted in poorer filter efficiency and more algal trouble in the basins, but altering the ratio of coagulant added in the primary and the secondary basins to 1 : 1 improved the filter influent and efficiency. When sodium aluminate below the normal lime application was used the final effluent contained too great a concentration of free carbon dioxide and had a tendency to be corrosive. R. E. THOMPSON.

Industrial wastes as manures. REGE.—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 14, 1926.

I.—GENERAL; PLANT; MACHINERY.

Enclosed continuous filter. J. F. Wait (Ind. Eng. Chem., 1926, 18, 295—297).—A perforated drum supporting a filter cloth revolves within a shell, fitted with external controls and sight-glasses, in which the liquid to be filtered is maintained by pressure at the required level. The drum contains hollow sections connected to the filtrate outlet and to a supply of air or vapour under pressure. A scraper removes the cake from the cloth and drops it into a draining hopper. The cake is dried on the cloth by air or vapour blown through it, or preferably by a circulation method in which the air is withdrawn with the filtrate and returned to the filter vessel in a closed circuit. The cake produced by this method is considerably drier than that formed in a leaf filter or by a centrifuge. The process is applicable to volatile liquids, such as petroleum distillates, and enables filtration to be carried on under vacuum or pressure at elevated temperatures, which is a great advantage in the treatment of viscous oils or liquids.

B. W. Clarke.

New method of conducting filtration tests. D. R. Sperry (Ind. Eng. Chem., 1926, 18, 276—279). —The vertical movement of a float, situated in a cylindrical vessel in which the discharge from an experimental filter-press is collected, actuates a pen which records automatically the time–discharge curve of the process on a cylinder rotated at one revolution per hour by clockwork. This reduces the time actually required for observation of filtration tests and gives the time–discharge curves plotted ready for use. It has been operated successfully in filtration tests on syrups, clay slips, varnishes, plating liquors, etc., and may be adapted to the study and control of large-scale filtering operations.

B. W. Clarke.

Fractionating column calculations. T. S. Carswell (Ind. Eng. Chem., 1926, 18, 294—295).—The height of the equivalent theoretical plate, *i.e.*, the length of packing required to produce equilibrium between the vapour and liquid in the fractionating column, is shown to be directly proportional to the radius of the rings used for packing and independent of the radius of the column. It varies with the nature of the material in the column according to the equation $H.E.T.P.=kMd/T$, where M is the average molecular weight of substances under distillation, T the temperature of distillation, d the density of backflow in the column at the distillation temperature. This is confirmed by experimental data from the distillation of alcohol–water mixtures, benzene–toluene mixtures, etc. B. W. Clarke.

See also A., April, 343, **Air bubble viscosimeter** (Barr). 345, **Adsorption of gases by activated charcoal at very low pressures** (Rowe). 346, **Theory of adsorption by carbon** (Ruff); **Adsorption by coconut charcoal of saturated vapours of pure liquids. Adsorption by coconut charcoal from mixed vapours** (Tryhorn and Wyatt). 347, **Sorption of vapours by alumina** (Munro and Johnson); **Quantitative adsorption analysis by Wislicenus' method** (Lorenz). 365, **Inactivation of catalysts during transformation of carbon compounds** (Zelinski); **Fourth report on contact catalysis** (Taylor). 377, **All-metal mercury-vapour pump** (Kaye); **Continuous dialysis or extraction apparatus** (Hanke and Koessler); **Wiegner's elutriation apparatus** (Gessner). 378, **Nephelometer and colorimeter** (Dold); **Turbidimeter** (Baylis).

Bituminous coal and coke for generating steam in a low-pressure boiler. Augustine, Neil, and Myler, jun.—See II.

Patents.

Centrifugal apparatus. E. C. Duhamel, and Comp. Gén. des Industries Textiles (E.P. 247,092, 6.8.25. Conv., 2.3.25).—Deposited solid matter is removed from the non-perforated bowl of a centrifugal machine, intermittently but without stopping the rotation, by means of a scraper operated like a lathe tool, *i.e.*, a small narrow knife is given a reciprocating axial motion the full depth of the bowl and is gradually fed radially into the deposit. The axis of the machine is preferably horizontal, the scraper is situated in the upper part of the bowl, and the detached material drops into a guide which deflects it outwards from the bowl. B. M. Venables.

Centrifugal machines and processes. Sharples Specialty Co., Assees. of L. D. Jones and A. U. Ayres (E.P. 233,327, 15.4.25. Conv., 1.5.24).*—The patent relates to a centrifugal apparatus in which the treated substance is discharged in dispersed form, and in which the atmosphere within the separator may be regulated to control the condition of the treated substance. The bowl, 1, is supported inside the closed casing, 10, and has outlets, 3, 4, which discharge into chambers, 8, 7. The pulley, 25, of the machine is driven by the pulley, 22, operated by an external motor. The mechanism for transmitting motion to the bowl comprises a part, *e.g.*, the hollow shaft, 26, which is supported firmly in its bearing, 27, and another part, *e.g.*, the spindle, 60, which is free to vibrate with the bowl during its rapid rotation. The shaft, 26, may be sealed by packings, 31, 32, with a ring, 33, between them providing a chamber, 34,

around the shaft. Passages, 36, provide communication between the chamber, 34, and a groove, 35, on the ring connected by a passage, 38, to a pipe, 39, whereby pressure in the chamber may be

FIG.1.

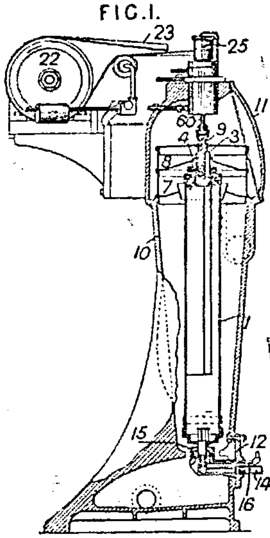


FIG.4.

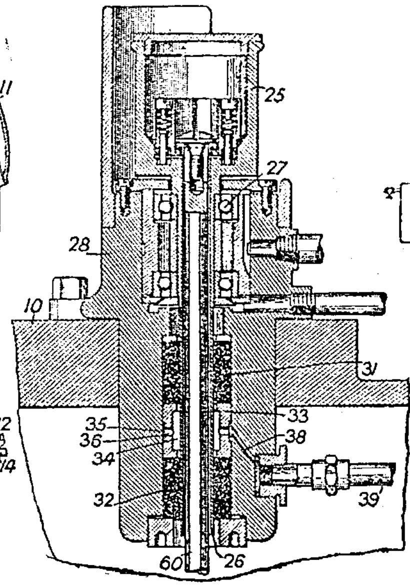


FIG.5.

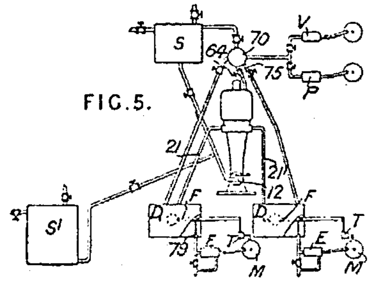
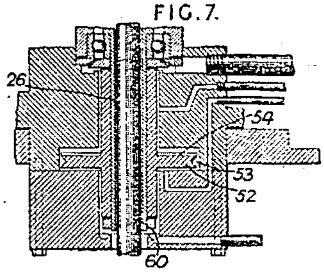


FIG.7.



regulated or the chamber may be supplied with liquid according to the working of the machine. A seal of the labyrinth type may also be used, or a liquid seal as shown in Fig. 7. A sleeve on the shaft carries a disc, 52, provided with impeller vanes, 54, to propel fluid in the chamber, 53, in a direction opposite to that in which the leakage pressure would tend to move it. Fig. 5 shows the connexions whereby the separator can be exhausted by the vacuum pump, *V*, or placed under pressure by the pump, *P*; or a gas may be supplied by the pump, *P*, and partly withdrawn by the pump, *V*; *S*, S^1 are supply tanks and *D* discharge tanks. Low pressure may be used to cause evaporation, or prevent foaming and oxidation, and higher pressure may be used to prevent escape of fumes or dissolved gases. An inert atmosphere may be used to prevent chemical change or the latter may be effected by a chemically active gas. R. B. CLARKE.

Machine [horizontal centrifuge] for separating liquids from solids. H. C. BEHR (U.S.P. 1,565,002, 8.12.25. Appl., 24.7.22).—A rotating horizontal or inclined shaft is fitted with two concentric conical screens which rotate in the same direction at different rates, the outer being the faster. The mixed liquid and solids to be treated are introduced axially at the small end of the cones and pass through the intermediate space towards the bases under the action of centrifugal force and of screw threads cut in the inner screen. The liquid passes through the outer screen, while the solid is withdrawn at the bases of the cones. Fan blades are attached to the shaft to force air from inside the inner screen through the solid so as completely to free it from liquid and also to assist the passage of the material through the apparatus. Baffles are also provided which distribute the material evenly on the screens and so prevent undue strain on the shaft.
T. S. WHEELER.

Centrifugal separator. W. C. LAUGHLIN, Assr. to LAUGHLIN FILTER CORP. (U.S.P. 1,572,612, 9.2.26. Appl., 2.6.25; cf. E.P. 235,916; B., 1926, 255).—A centrifugal bowl with horizontal axis has concentric discharge throats at each end, and a peripheral discharge at some point between the ends surrounded by a collector.
B. M. VENABLES.

Tube mills for grinding or crushing. WICKING'SCHE PORTLAND-CEMENT & WASSERKALKWERKE, Assees. of A. ANDREAS (E.P. 241,174, 14.9.25. Conv., 11.10.24).*—The apparatus comprises a

FIG.1.

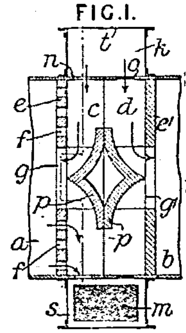
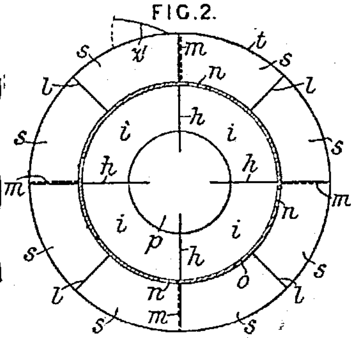


FIG.2.



coarse-grinding chamber and fine-grinding chamber with an intermediate transferring and screening device. The partly ground material passes through the apertures, *f* (Fig. 1), in the end wall, *g*, of the coarse grinding chamber into the transfer chamber, *c*, and through the ports, *n*, into the annular sifting chamber, *s*, which is shown (see Fig. 2) divided into eight compartments by alternate impervious

walls, l, and screens, m. Material that passes through the radial screens, m, emerges from the sifting chambers through ports, o, into the right-hand side, d, of the transfer chamber, and, striking the deflector, p, enters the fine-grinding chamber through the central aperture, g^1, in the end wall. The material that is too coarse to pass the screens, m, returns through the ports, n, to the left-hand side, c, of the transfer chamber and is deflected back into the coarse-grinding chamber through the central aperture, g. The screens may be changed or cleaned through dust-tight flaps, z^1; if desired the screens and walls, m and l, may be curved as blades and the two transfer spaces, c and d, may be separated by an impervious wall. B. M. Venables.

Ball and ring and roller and ring mills for grinding and crushing. C. E. V. Hall (E.P. 247,756, 10.3.25).—The mill is of the pendulum-roller and ring type, the pendulum arms rotating in ball bearings carried by a spider. The spider and driving pulley are mounted on a sleeve shaft running on a fixed central shaft.
B. M. Venables.

Grinder and pulveriser. H. J. Shelton (U.S.P. 1,572,692, 9.2.26. Appl., 17.7.22).—The housing of the apparatus comprises a main casting and a side casting hinged to it. A grinding ring rotates within the main casting and a pair of grinding rolls make contact with the ring at diametrically opposite points, each being mounted on a shaft carried by an arm pivoted on the side casting. The grinding rolls are kept in position against the grinding ring by a spring. H. Moore.

Ore-grinding mill. W. N. Beyerle (U.S.P. 1,573,032, 16.2.26. Appl., 4.3.24).—A number of balls ride in a circular groove. A weighted upper member with a similar circular groove rides on the balls. The material to be ground is introduced through a hopper in the centre of this upper member and when sufficiently fine emerges through vertical screens, surrounding the outer circumference of the grooves, into an annular space. The portions of the mill are held together so as to yield sufficiently when the upper member is rotated.
W. N. Hoyte.

Extraction and recovery of volatile liquids. E. A. Ironside (E.P. 246,930, 8.11.24).—In an apparatus for extraction of greases etc. by means of a volatile solvent, the solvent is removed from both the extract and from the residual material by means of steam or hot air, the bulk of the solvent being recovered in a condenser, connected with a decanter in which condensed water is separated. At the end of the distillation stage, the vapours which are weak in solvent are passed through an adsorbent, from which the solvent is subsequently removed by heating and recovered in the condenser.
B. M. Venables.

[Dust] containers and means for emptying them. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 247,744, 24.2.25).—A container filled with dust (coal, cement, dyes) is emptied by means of one or more rotating jets of air within the lower part of the container. The container is made air-tight so that the dust, having been loosened by the air jets, is expelled by the accumulated air pressure. B. M. Venables.

Adsorbent material [from silica gel]. C. S. Teitsworth, Assr. to Celite Co. (U.S.P. 1,570,537, 19.1.26. Appl., 27.3.24).—Powdered diatomaceous earth, pumice, or other solid porous substance is mixed with sodium silicate solution, and silica gel is formed by addition of hydrochloric acid. The gel which contains the earth (2—20% on the weight of the gel) dispersed through it has an adsorbent power very much greater than that of ordinary silica gel. For example, it will adsorb 83% of its weight of water from air saturated at 20°. Other inorganic gels such as aluminium hydroxide gel may be used.
T. S. Wheeler.

Heating gases [air] for drying. W. A. Darrah (U.S.P. 1,571,575, 2.2.26. Appl., 25.4.23).—An apparatus for heating indirectly gases such as air which are to be used in dryers, consists of a series of combustion chambers in which a fuel such as oil is burned. The walls of these chambers are formed of a highly conducting material, such as carborundum, while those of flues leading from them which are less highly heated are of metal. The air to be heated is drawn, in counter-current to the flue-gases, through conduits in contact with the combustion chambers and flues. All the passages and flues are tortuous and are fitted with baffles so that eddies are set up. The walls between the air conduits and flues are made of highly conducting material, but walls of material of low heat conductivity, such as firebrick, are also provided in the air passages, and are heated by radiation from the conducting walls. A 70—90% thermal efficiency is attained in practice.
T. S. Wheeler.

Combined spreading and scraping mechanism for distillation apparatus. W. M. Grant, Assr. to Illinois Anthracite Corp. (U.S.P. 1,569,478, 12.1.26. Appl., 8.11.23).—The mechanism comprises a moving hopper which feeds material into a retort or oven, and means for levelling and smoothing the charge as it is fed in.
A. B. Manning.

Fractional condensation. J. E. Bell, Assr. to Sinclair Refining Co. (U.S.P. 1,573,129, 16.2.26. Appl., 23.3.22).—The vapours to be condensed are brought in contact with pipes containing a circulating cooling medium, the temperature of which is regulated by introduction of fresh medium.
W. N. Hoyte.

Filtering process. H. S. Coe, Assr. to Cycle Co. (U.S.P. 1,574,558, 23.2.26. Appl., 26.1.20. Renewed 29.10.25).—A number of filtering tanks, each with a horizontal filter bottom arranged so that a settling action also takes place, are arranged in a series so that the solid matter (thick pulp) collected on any one (say the first) is continuously

removed and mixed with the filtrate from another tank (say the third) and re-filtered in another tank (say the second). B. M. VENABLES.

Apparatus for effecting catalytic gas syntheses under pressure. G. L. E. PATART (F.P. 598,966, 25.5.25).—Reacting gases pass under pressure in the same direction through a horizontal tube containing catalytic material. L. A. COLES.

Production of catalysts for hydrogenation processes. J. D. RIEDEL A.-G. (G.P. 424,067, 24.9.20).—Nickel or cobalt salts, or mixtures of these with copper salts, are reduced in a pure, non-basic liquid medium which is stable at high temperatures, such as molten naphthalene, or toluene or acetone, or their hydrogenation products, such as tetrahydronaphthalene, methyl*cyclo*hexane, or *iso*propyl alcohol, and the catalyst is subsequently separated from the medium and from volatile reaction products by distillation. L. A. COLES.

Preventing the deposition of adherent crystals [scale] upon metal surfaces. ANTISCALE A.-G. ZUR VERWERTUNG INDUSTRIELLER PATENTE (F.P. 599,188, 8.6.25. Conv., 3.7.24).—The walls of vessels containing solutions from which crystals may separate are subjected to the action of a constant, variable, or intermittent magnetic field, whereby weak electric currents are induced in the liquid. L. A. COLES.

Centrifugal separator. W. C. LAUGHLIN, Assr. to LAUGHLIN FILTER CORP. (U.S.P. 1,572,611, 9.2.26. Appl., 21.6.24).—See E.P. 235,916 ; B., 1926, 255.

Producing gels for catalytic and absorbent purposes. W. A. PATRICK (U.S.P. 1,577,186, 16.3.26. Appl., 28.2.20).—See E.P. 159,508 ; B., 1922, 812 A.

Preparing catalytic agents. W. A. PATRICK, Assr. to SILICA GEL CORP. (U.S.P. 1,577,187–8, 16.3.26. Appl., 18.11.22).—See E.P. 212,034–5 ; B., 1924, 360.

Drying apparatus. O. SÖDERLUND, T. BOBERG, and N. TESTRUP, Assrs. to TECHNO-CHEMICAL LABORATORIES (U.S.P. 1,577,545, 23.3.26. Appl., 9.6.24).—See E.P. 219,792 ; B., 1924, 815.

Tanks for treatment of liquids or solids mixed with liquids. R. O. STOKES (E.P. 248,679, 3.10.24).

Condensing and/or cooling apparatus. E. H. BECKETT (E.P. 248,872, 24.12.24).

Liquid-cooling towers. O. SORGE (E.P. 249,420, 6.10.25. Conv., 28.5.25).

Bricks containing metals or metal oxides (G.P. 422,715).—See IX.

Electrical precipitation (U.S.P. 1,574,237 and 1,575,165).—See XI.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Influence of moisture on the spontaneous heating of coal. J. D. DAVIS and J. F. BYRNE (Ind. Eng. Chem., 1926, 18, 233—236).—The spontaneous heating of Pittsburgh and Sharon coals was examined in an adiabatic calorimeter, the initial temperature being 70°. The coals as received, containing 2·2% and 11·8% of moisture respectively, did not heat in dry oxygen ; in oxygen saturated at room temperature the coals heated or cooled depending on the rate of circulation of oxygen and the resulting rate of moisture evaporation. Curves are given showing the heating of the coals with varying degrees of moisture in dry oxygen. With coals dried at 140° for 22 hrs. the heating proceeded slowly from 70° to 97°, where it tended to stop, due either to the complete removal of moisture or to a change in the colloidal state of the coal. It is concluded that it is better to store coal moist than dry. A. C. MONKHOUSE.

Indirect determination of the calorific value of coal. J. GEIDEL and W. REHWINKEL (Chem.-Ztg., 1926, **50**, 116—117).—A method of calculating the calorific value of a coal is described, based on the constancy of the calorific value of the pure coal substance in all coals of the same origin. Knowing this value (R) for a particular mine or field, the calorific value (H) of a coal from that mine or field can be calculated from a determination of its ash (A) and moisture (W), thus: $H=R[100-(A+W)]/100-6W$, both H and R being net values. The value of R is found from one, or better, several exact calorimetric determinations for the coal mine or field in question. The error of the method does not in general exceed 1%. The results obtained for a number of coals of various origins are tabulated and compared with the directly determined calorimetric values, as well as with the values given by other indirect methods. A. B. MANNING.

Theoretical and recorded pressures in oxygen bomb determinations [of calorific value]. M. J. BRADLEY, C. Z. ROSECRANS, and R. M. CORBIN (Ind. Eng. Chem., 1926, **18**, 307—309).—In order to determine the limits of weight of fuel and initial oxygen pressure which can be safely used, experiments were made with crude petroleums, coals, benzene, kerosene, and benzoic acid; the pressures developed within the oxygen bomb during combustion were determined and photographic records made. A heat loss of 50% is assumed before the maximum temperature is attained. Rate of combustion and therefore the maximum pressure may be controlled by the condition of the fuel and the size and shape of the containers in the bomb. For kerosene and crude benzol, the weight of material should not exceed 1·5 g., and the initial oxygen pressure should be as low as possible. By accurately determining the volume of the bomb and the Bourdon gauge, and leading 100 c.c. of the gases produced on combustion into an Orsat gas apparatus, and determining

the carbon dioxide, the carbon content of the original material may be calculated. Further, by cooling the bomb to the original temperature the loss in volume due to combustion of hydrogen may be directly read from the gauge. D. G. Hewer.

Sampling and examination of mine gases and natural gas. G. A. Burrell, F. M. Seibert, and G. W. Jones (U.S. Bur. Mines, Bull. 197, 1926, 108 pp.).—A revision of Bull. 42, in which the latest laboratory methods for the analysis of mine and natural gases are described, together with the latest types of apparatus (cf. Burrell and Seibert, B., 1913, 1096; 1914, 808; 1915, 267; 1916, 947). A large number of illustrations are given of apparatus designed for special purposes, *e.g.*, for the complete and accurate analysis of mixtures containing small amounts of different constituents and of mixtures rich in methane, hydrogen, and carbon monoxide; for the rapid determination of methane and carbon dioxide in mine air; and for the determination of carbon dioxide and oxygen in connexion with ventilation problems. A. R. Powell.

Value of bituminous coal and coke for generating steam in a low-pressure cast-iron boiler. C. E. Augustine, J. Neil, and W. M. Myler, jun. (U.S. Bur. Mines, Tech. Paper 367, 1925, 45 pp.).—By-product coke, Pittsburgh high-volatile lump coal, and Lower Kittanning medium-volatile coal having calorific values of 11,720—11,890, 13,150—13,750, and 12,970—13,570 B.Th.U./lb. respectively, were burned in a hand-fired low-pressure cast-iron boiler of a size suitable for heating large buildings with the object of comparing the relative steaming values of the fuels and of separating the heat losses in order to determine the effect of the method of firing and of structural changes in the boiler when operated at ordinary capacities. The firing period was varied from 60 to 20 min. to suit the different rates of steaming, which were from 50% to 125% of the rated capacity of the boiler. At low rates of steaming, the steaming values (*i.e.*, thermal efficiency × calorific value) of the bituminous coals and of the coke were about equal, but at high rates, that of the coke was about 90% of the value of the bituminous coals. The thermal efficiencies obtained using coke, Pittsburgh coal, and Lower Kittanning coal varied from 66% to 76%, 65% to 74%, and 66% to 72% respectively, Pittsburgh coal showing the lowest and coke the highest efficiency at low rates of steaming. The heat transferred to water per square foot of heating surface per hour varied from 3400 to 10,000 B.Th.U. Other things being equal, the nearer to the fuel bed the secondary air is supplied and the more thoroughly it is mixed with the rising combustible gases, the more rapid and efficient will be the combustion. Wing walls leaving a vertical slot in the centre line of the boiler are rather more efficient as a mixing device in the combustion chamber than chequer-work. The use of such a mixing device also helps materially to reduce smoke. The pressure drop of air through the fuel bed was about the same for coke and Pittsburgh coal and was about half that required for equal ratings with Lower Kittanning coal, which ignited less readily, burned more unevenly, and required more frequent attention. A. W. Hothersall.

Determination of unburnt carbon from the analysis of flue-gases. W. R. Chapman (Fuel 1926, 5, 128—129).—The amount of unburnt carbon lost during an industrial heating operation can be calculated from the analysis of the flue gases and of the coal, using the following equation:—% of carbon unburnt=

$$100+37\cdot5\frac{\epsilon}{a}-\frac{1}{a}\left[\frac{(r+s+v)(1140\beta+42\cdot8\delta-142\cdot5\gamma)}{100-4\cdot8r-2\cdot9s-4\cdot8t+0\cdot9u+2\cdot8v}\right]$$

where a, β, γ, δ, ϵ represent the percentage in the coal, calculated on the ash-free, dry basis, of C, H, O, N, and S respectively, and r, s, t, u, v represent the percentage in the flue-gases of CO_2, CO, O_2, H_2, and CH_4 respectively. A. W. Hothersall.

Determination of the degree of swelling of coking coals. M. Dolch (Brennstoff-Chem., 1926, 7, 69—73).—A simple apparatus is described for determining the apparent volume of a sample of coke by displacement, using fine lead shot as a filling material. It is suggested that the determination should be included in the course of the ordinary examination of coking coals, following the coking test, the same coke sample being afterwards used for the ash determination. At the same time the density of the coal is determined on a number of pieces of suitable size, or, if data are available, by calculation from the ash content. The degree of swelling is expressed by the ratio coke volume/coal volume. The results for 7 gas coals are tabulated. In general the degree of swelling determined on a finely powdered coal differs considerably from that obtained by coking a single piece of the same coal (cf. Krönig, B., 1926, 160). A. B. Manning.

Research on low-temperature carbonisation at the Sarre mines. J. St.-Claire Deville (Chim. et Ind., 1926, 15, 163—172).—Experiments have been carried out with two plants, one a small retort of the Salerni type of 100 kg. capacity, the other a setting of two larger Salerni retorts, each of 15 tons throughput per day. On carbonising a washed fine coal high in moisture practically the whole of the water is evolved before oil and gas begin to appear. Once started, however, the evolution of the tar proceeds rapidly (95% within an hour with the smaller apparatus) and is complete when the temperature has reached 500°. Raising the temperature of the semi-coke further yields only a gas rich in hydrogen, and some ammonia. Details of working and some difficulties met with in the operation of the larger plant are described. The washed fine coal, containing 8% of ash on the dry material and 15—20% of moisture, yields 50 kg./ton of a heavy tar, high in tar acids and poor in paraffin wax, and nearly 100 m.³ of a rich gas containing 40 g./m.³ of crude light oil, which, on refining, gives 75% of its volume of a good quality motor spirit. A schist containing bands of vitrain (60—65% of ash, 16—18% of volatile matter, and 2% of moisture)

yielded 40—50 litres of a light tar with 28% of phenols, and rich in lubricating oils and solid paraffins. The economic aspect of low-temperature carbonisation is briefly dealt with; the process can only be economically successful when applied to fuels of poor quality, similar to those mentioned above.
A. B. MANNING.

Combustibility of blast-furnace coke. R. A. SHERMAN and S. P. KINNEY (Fuel, 1926, 5, 98—105).—The combustibility of coke as determined by exploring the hearth zones of blast furnaces and of an experimental furnace by sampling the gases at intervals across the hearth (cf. Sherman and Blizard, B., 1923, 436 A; Royster and Joseph, B., 1924, 470) is not greatly influenced by the coking time, specific gravity, porosity, volatile matter, or the coking temperature. The size of the pieces, however, has a marked effect on the combustibility. The size of the pieces of coke which reach the tuyères of a blast furnace will be determined by the resistance of the coke to impact and abrasion. Such variations as were found in similar sizes of various cokes were so slight that it is improbable that they would make any material difference in furnace operation. There is no definite relation between the character of the coke and its combustibility in carbon dioxide as determined by Perrott and Fieldner (Amer. Soc. Testing Materials, June, 1923). The available data show considerable variations in the combustibility of various cokes in carbon dioxide, but they show no apparent relation to the actual operating data. The correlation of laboratory tests on the combustibility of coke in air and in carbon dioxide with actual furnace conditions is difficult if not impossible.
A. W. HOTHERSALL.

Coal gas condensation. T. H. PRATER (Gas J., 1926, 173, 544—547).—Coal gas condensation is reviewed with special reference to the hydrocarbon content of the cooled gas. Shock cooling in water-cooled condensers yields tar of higher water content than slow cooling in atmospheric condensers, and when the respective tars are distilled, although on the dry basis they contain similar proportions of "light oil," the water-cooled condenser tar yields more "light oil" distillate up to 170° because of the enhanced "steam distillation effect" of its greater water content. Rapid condensation is more efficient in the removal of naphthalene than slow condensation over the same temperature range. Laboratory experiments on the washing of gas at various temperatures with tar from different sources were made. Washing at 80°, except with tar previously distilled to 170°, materially increased the hydrocarbon content of the gas. Below 80°, the results were dependent on the "light oil" content of the tar; under favourable conditions an improvement was effected but generally the process was detrimental.
S. PEXTON.

Elimination and recovery of phenols from crude ammonia liquors. R. M. CRAWFORD (Ind. Eng. Chem., 1926, 18, 313—315).—At three coke-oven installations in America the phenols are now extracted from crude ammonia liquor by washing with benzol. This operation is performed in two large towers in series, the benzol being sprayed upwards from the bottom through the descending ammonia liquor with which the towers are filled. The phenolated benzol is drawn off from the top of the second tower and is sprayed through caustic soda in two similar but smaller towers. The sodium phenoxide solution is circulated through one tower only till saturated. It is then drawn off and worked up as usual, the weaker phenoxide from the second tower being transferred to the first and fresh caustic soda added to the second. A portion of the benzol is continuously by-passed from the circuit and passed through sulphuric acid to remove pyridine bases. The crude phenol finally produced contains about 50% of phenol and 26% of cresols. With liquor containing 2 g. of phenols per litre 16·6 lb. of crude phenol are recovered per 1000 gals. with a loss of 5 lb. of benzol. The process removes difficulties in the disposal of ammonia-still effluent caused by phenols.
C. IRWIN.

Properties of typical crude oils from the producing fields of the Western Hemisphere. A. J. KRAEMER and L. P. CALKIN (U.S. Bur. Mines, Technical Paper 346, 1925, 43 pp.).—A compilation of the more important chemical and physical characteristics of crude oils produced in Canada, Mexico, Trinidad, Venezuela, Argentina, Colombia, and Peru. For comparison the analyses of typical crude oils from the chief producing districts of the United States are also given. Considered broadly the Canadian oils have a high gasoline and low sulphur content. The Mexican oils are divided into two classes according to the geographical position of the fields and known as Northern heavy oil and Southern light oil. These asphaltic crudes are all highly sulphurous. Trinidad oils show variation between different fields, the sulphur varying from 0·3% to 2·6% and gasoline from 11 to 50%. Although the Argentine oils are similar chemically two samples show considerably less volatile components than the other two. Two samples each from Venezuela, Colombia, and Peru are tabulated, one from Peru showing a sulphur content of 0·02% and containing 43% of gasoline.
S. BOWMAN.

Industrial requirements for dry cleaner's naphtha. L. E. JACKSON (Ind. Eng. Chem., 1926, 18, 237—238).—The necessary properties of the naphtha are discussed, and the following specification is proposed:—Free from water; water-white colour; negative doctor test; not more than 2% of unsaturated compounds; distillation range 138—204°; acidity nil; aromatic content nil; sweet odour.
W. N. HOYTE.

New process for regeneration of spent decolorising powder. L. GURWITSCH (Kolloid-Z., 1926, 38, 247—248).—A new method for regenerating decolorising powder used in the mineral oil industry, which avoids its removal from the filter, consists of extraction with a mixture of benzene and alcohol, and subsequent heating to about 150°. The extrac-

tive power of a solvent depends on its heat of wetting. Alcohol has a comparatively high heat of wetting (17·2 cal./g. against "Floridin S"), but is not a good solvent for the impurities it is desired to extract. On the other hand benzene is a bad "wetter" but a good solvent. The combination of the two thus effects satisfactory extraction. By the application of this method the same sample of "Floridin" was used for about 30 operations without any marked diminution in its decolorising power.

N. H. HARTSHORNE.

Machinery lubricating oils. J. SWOBODA (Petroleum, 1926, 22, 247—253).—The most important qualities of a lubricant are its oiliness, or property of clinging to the lubricated surface, and its viscosity, or internal friction. The greater the viscosity the less the expenditure of lubricant, and the less it is liable to be displaced from between the bearing surfaces, but the greater is the friction. The oil should have minimum viscosity consistent with sufficient oiliness to ensure adherence to the rubbing surfaces. For greater speeds of rubbing surfaces oils of lower viscosity are required. The oil must resist change from the action of the air and high temperatures, must have a reasonably high flash point, must be quite free from inorganic acidity, and not have excessive organic acidity. Its boiling point should be high and its cold test low. The author discusses the properties of the various animal and vegetable oils, which possess greater oiliness than mineral oils, as regards their suitability for use in compounding mineral oils, and the requirements in lubricating oils for engines and machines of different categories.

H. MOORE.

Lubrication problem from the thermodynamic-molecular standpoint, and the measurement of the efficiency of lubricants. R. VON DALLWITZ-WEGNER (Kolloid-Z., 1926, 38, 193—208).—The theory of lubrication is discussed and some methods of measuring the efficiency of lubricants are described. Bearing metals have a high molecular cohesion pressure of the order of 100,000 atm., while lubricants have a relatively low cohesion pressure of 300—400 atm. In a lubricating layer part of the cohesion pressure, K, of the lubricant is converted into a wetting pressure, K_b, on account of molecular attraction, and generally the extent of this conversion may be obtained from the relation, $K_b/K=\cos\theta$, where θ is the "marginal angle" of the lubricant, *i.e.*, the angle between the edge of a drop of the lubricant when placed on a plane surface of the metal, and that surface. Other things being equal the cosine of the marginal angle is a measure of the efficiency of the oil, for this depends on the wetting pressure, which determines the thickness of the lubricating layer. The marginal angle varies with the metal lubricated and can thus be used to find the most suitable metal for a particular oil. If it is desired to increase the viscosity of a lubricant, the sphere of action of the molecules must be increased. Since $\lambda=3a/K$, where λ is the sphere of action, a the surface tension, and K the cohesion pressure, this may be done by increasing a or decreasing K. The former process appears to be unsatisfactory, *e.g.*, tar oils have a high surface tension and a relatively small marginal angle, but are not good lubricants. Apparatus is described for measuring the thickness of the lubricating layer between a rotating shaft and its bearing, and for determining the marginal angle of an oil from measurements of the surface tension and wetting pressure, since $\cos\theta$ = wetting pressure/surface tension.

N. H. HARTSHORNE.

***X*-Ray spectrographic investigations on lubricants.** J. J. TRILLAT (Compt. rend., 1926, 182, 843—846).—Bragg's hypothesis (Nature, 1925, 115, 266), that the origin of greasiness is to be found in the laminar structure which certain substances assume under pressure, is confirmed by the *X*-ray spectrographs of thin layers of greases formed under gentle pressure. This structure, which is destroyed by fusion, is probably present in all lubricants; it was feebly apparent in some oils. The laminæ of triglycerides are bimolecular; those of saturated hydrocarbons are unimolecular. It is considered that molecular orientation, favoured by the presence of certain active groups, takes place at the lubricated surface; thus in mixtures of fatty substance with mineral oils the former attaches itself to the metal and undergoes orientation, forming a surface of methyl groups over which the hydrocarbon molecules can slide very easily. The analytical significance of the *X*-ray spectrographs is alluded to.

S. K. TWEEDY.

See also A., April, 341, **Pressures developed on explosion of gaseous mixtures at high densities** (DAVID). 375, **Absorption of carbon monoxide by solutions of cuprous chloride** (MOSER and HANIKA). 380, **Microscopic structure of pit-coals** (DUPARQUE). 381, **Hydrogenation of organic substances at high temperature under pressure** (KLING and FLORENTIN). 420, **Determination of unsaturated in presence of saturated and tricyclic hydrocarbons** (NAMETKIN and BRÜSSOFF).

Carbonising sulphite-cellulose waste liquor with mordant salts. SCHWALBE.—See V.

PATENTS.

Drying materials [coal with waste furnace gases]. M. D. JONES, Assr. to FULLER FUEL CO. (U.S.P. 1,568,738, 5.1.26. Appl., 18.3.24).—Waste furnace gases at a high temperature and carrying a large proportion of moisture are mixed with cool air of low moisture content so that the temperature and humidity of the gases are reduced, and the mixture is passed over the fuel to be dried, for example, coal which is to be pulverised. All risk of ignition is avoided and rapid and uniform drying is obtained.

T. S. WHEELER.

Manufacture of combustible briquettes. J. BEAUDEQUIN (E.P. 225,849, 2.12.24. Conv., 3.12.23).—Carboniferous material, such as coal waste, is finely ground, compressed into briquettes, and charged into a horizontal tube externally heated to 300°. Each briquette is separated from its

neighbour by a division plate. The briquettes are slowly pushed along, the rate being adjusted to obtain satisfactory binding. The tube protrudes out of the furnace to form a cooling chamber for the briquettes before these are discharged. S. Pexton.

Dehydrated lignite. E. P. Schoch (U.S.P. 1,574,174, 23.2.26. Appl., 18.8.24).—Lignite is dehydrated by heating with a hydrocarbon oil, the moisture being displaced by the oil, which latter is subsequently removed. A. C. Monkhouse.

Coking retort ovens. Koppers Co., Assees. of J. van Ackeren (E.P. 227,087, 29.10.24. Conv., 4.1.24).—In a regenerative vertical retort oven, the long vertical heating flues are supplied with hot fuel gas and hot secondary air at the middle as well as at the top. The products of combustion leave the vertical flues on one side of the retort at the bottom and pass up similar flues on the opposite side prior to entering one of a pair of regenerators. During the second part of the cycle the flows of fuel gas and products of combustion are reversed. S. Pexton.

Coking retort oven. J. van Ackeren, Assr. to Koppers Co. (U.S.P. 1,570,870, 26.1.26. Appl., 5.8.21).—In a coke-oven battery comprising coking chambers and vertical combustion flues, the latter are disposed in two groups, and can be put into communication with inflow and outflow groups of regenerators on each side and at opposite ends of the battery. A producer-gas supply is provided for heating the battery, with reversing valve connexions between the gas supply and some of the regenerators of each group on both sides of the battery. On each side of the battery are waste-gas tunnels with valves for establishing communication between the outflow regenerators on both sides with the corresponding waste-gas tunnels.
A. B. Manning.

Coke oven with vertical heating flues. P. Chomé and L. Defossez (E.P. 247,646, 17.11.24).—A coke oven with horizontal coking chambers is provided with vertical flues which can be heated either from the top or from the bottom, the method of heating being reversed at intervals in order to attain more uniform carbonisation. The oven can be built either without or with regenerators and, in the latter case, is designed for heating either with rich gas or with poor gas by using gas regenerators as well as air regenerators. All the vertical heating flues are connected at their lower ends with two horizontal flues running below the oven, and at their upper ends with another pair of horizontal flues in the oven wall. Each of the upper and lower horizontal flues is connected to the regenerators or to the reversing device, the upper by one or more vertical conduits situated in the oven wall, the lower directly. When using a rich gas, this is admitted to burners in the top or the bottom of the vertical flues according to the method of heating, air from the regenerators being supplied through the corresponding pair of horizontal flues, and the products of combustion escaping by the opposite pair. When using poor gas, the latter is heated in the regenerators and passes thence to the heating flues by one of the upper or lower horizontal flues, air being supplied by the other upper or lower flue respectively. Other modifications of the heating arrangement are possible, utilising supplies of both poor and rich gas.
A. B. Manning.

Distillation of carbonaceous material. M. Fränkl (E.P. 238,899, 24.8.25. Conv., 23.8.24).—A retort of rectangular section has nostril holes distributed throughout the lower portion of its two major walls. Preheated air admitted to the retort, via a regenerator and the nostrils holes on one side, consumes part of the carbonised fuel, and heats the charge, the combustion products leaving by way of the nostrils and regenerator on the opposite side. When the fuel bed has attained the desired temperature the supply of air is stopped, and a non-oxidising gas (which may be the gas produced by the process), introduced into the bottom of the retort, is caused to traverse the charge and distil the coal entering at the top. When the temperature of the fuel bed has fallen materially the stream of inert gas is stopped and air is again introduced, but in a reverse direction to that followed previously. S. Pexton.

[Liberation of hydrogen and methane in the] distillation of coal. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude (E.P. 243,665, 2.4.25. Conv., 28.11.24).—A part of the hydrogen and methane ordinarily retained in the coke after the distillation of coal is recovered by passing nitrogen, air, or combustion gases through the retorts or ovens at the end of the distillation process. When nitrogen is used the mixture of hydrogen, methane, and nitrogen obtained may be mixed with the other gases from the distillation process, and the whole mixture of gases so obtained may be subjected to a partial liquefaction or other separation process for the extraction therefrom of hydrogen and nitrogen for use in the synthesis of ammonia. A. B. Manning.

Distillation of carbonaceous material. O. H. Hertel (E.P. 247,639, 13.11.24).—Coal, shale, or other carbonaceous material is distilled in a slightly tapered vertical retort, 12—18 in. in width, provided with means for internal heating (in addition to the usual means for external heating) placed within one or more steel tubes extending vertically through the retort. These tubes are of non-circular cross section and can be rotated, thereby exerting a lateral pressure on the charge of material and producing vertical passages for the escape of gas. The retort works intermittently. A movable piston actuated by hydraulic pressure permits compression of the charge after filling the retorts, and also aids in the discharge. The bottom of the retort can slide vertically and when raised closes the retort; when lowered it permits ejection of the charge through two inclined discharge shoots. The charge on ejection divides into two portions along the line of the gas passages, each portion being discharged through the corresponding shoot. This division of the charge

may be aided by separating blades disposed vertically within the retort. A. B. Manning.

Manufacture of carbon for decolorising and deodorising and other purposes. Artificial Coal Co. (Hamon Process), Ltd., and L. le W. Hamon (E.P. 246,954, 2.12.24).—On account of its self-bonding properties paper pulp waste (the fine fibres not retained by the screen of the paper-making machine, and subsequently recovered in a filter-press) forms a suitable material for manufacturing decolorising charcoal. Lime is added as a spacing material and the mixture carbonised and washed with acid. Less lime and therefore less acid are used than in similar known processes because of the fibrous structure of the paper pulp. A mixture of peat or other carbonaceous material of suitable dryness may be mixed with the paper pulp waste and treated in the same way. S. Pexton.

[Water-]gas manufacture. F. W. Steere (E.P. 247,803, 19.5.25).—In a water-gas plant consisting of a generator and superheater, the steam for the run is obtained by admitting water in a finely-divided spray at the top of the superheater during a " down " run and at the base of the generator during an " up " run. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 233,038, 109,323, and 21,028 of 1892.) A. C. Monkhouse.

Gas producer. J. F. Rogers, Assr. to Wellman-Seaver-Morgan Co. (U.S.P. 1,572,040, 9.2.26. Appl., 26.12.18).—A rotary producer with an independently rotating grate is provided with mechanism for automatically controlling the relative rates of rotation and causing one part to rotate more quickly than the other. S. Pexton.

Gaseous fuel production. J. R. Rose, Assr. to J. Harris (U.S.P. 1,573,524, 16.2.26. Appl., 28.5.23).—Water-gas is enriched by passing it through a mixing chamber in which a regulated quantity of a liquefied hydrocarbon gas is added to it. The latter should possess a vapour tension between 25 lb./sq. in. and 100 lb./sq. in. at atmospheric pressure and ordinary temperatures. A. B. Manning.

Utilisation of ferruginous limestone [in coke ovens and gas producers]. L. Schneider (F.P. 596,764, 22.7.24).—An intimate mixture of ferruginous limestone with pulverised coal and scrap iron or waste iron oxides and a suitable proportion of manganese is burnt in a coke oven or a producer. The sulphur in the coal is almost completely combined with the iron, and the gas evolved is almost free from sulphur. The highly ferruginous coke is used for the production of high-grade steel. B. W. Clarke.

Catalysts for the hydrogenation of oxides of carbon. G. Patart (F.P. 599,588, 25.5.25).—The catalytic material consists of zinc chromate, tungstate, vanadate, or manganate, or similar compounds which form basic salts containing at least two equivalents of zinc to one equivalent of the acid radical. L. A. Coles.

[Production of] hydrogen. G. Cicali (F.P. 599,614, 15.6.25. Conv., 16.2.25).—Water-gas is freed from carbon dioxide and sulphur and similar impurities. It is then compressed, cooled, and freed from carbon monoxide by cooling with liquid nitrogen. C. Irwin.

Obtaining volatile products by distillation [*e.g.*, of fuel] with superheated steam in a closed cycle. Le Gaz Industriel (Soc. Alsacienne des Anneaux Raschig et le Gaz industriel réunis) (F.P. 599,615, 15.6.25).—Gases produced by distilling material with superheated steam are cooled as they leave the retorts to a temperature sufficiently low to cause deposition of the higher-boiling products, and the deposited material is separated from the residual gases by suitable means. L. A. Coles.

Gas washers. Kirkham, Hulett and Chandler, Ltd., and S. Hersey (E.P. 246,978, 3.1.25).—A series of inverted cones built around a vertical rotating shaft raise water through the annular spaces between the cones and spray it by centrifugal force through the gas. The efficiency of washing is improved by gradating the depth of the cones so that the shortest lies on the outside, and the planes of rotation of the upper and lower edges of each cone lie between those of the next inner cone. The upper edge of each cone is perforated and, to increase the water-lifting power of the rotors, spiral vanes are provided in the annuli between the cones. S. Pexton.

Purification of gases [from iron carbonyl]. J. Y. Johnson. From Badische Anilin- & Soda-Fabr. (E.P. 247,050, 21.5.25).—Gas to be purified from iron carbonyl vapour is mixed with sufficient air and passed over an active contact material, such as charcoal or silica gel, when the carbonyl is oxidised with the deposition of iron oxide in the adsorbent, which when fouled is regenerated by acid washing. Alternatively the gas without oxygen may be passed over the adsorbent to remove the iron carbonyl by adsorption, and the adsorbent regenerated intermittently by air treatment and acid washing. Other substances, *e.g.*, hydrogen chloride, may be used instead of oxygen to decompose the iron carbonyl with formation of a non-volatile compound. S. Pexton.

Gas calorimeter. R. G. Griswold, Assr. to Doherty Research Co. (U.S.P. 1,572,283, 9.2.26. Appl., 3.7.22).—The gas in burning heats a current of water which at the same time displaces the gas from its container, so that the volume of water heated is proportional to the volume of gas burnt. The rise in temperature of the water is registered in units corresponding to the calorific value of the gas. A. B. Manning.

Distillation of oil-bearing materials. L. de Hernandez (E.P. 247,658, 19.11.24).—The material is distilled in a cylindrical retort supported by annular brickwork. The heating gases pass around the retort and also through a channel through its centre.

The distillates are removed at different levels and pass through a cooler; the lighter fractions pass through a purifier and dehydrating chamber to the top of a fractionating column. A. C. Monkhouse.

Cracking hydrocarbon oils. Sinclair Refining Co., Assees. of E. C. Herthel and H. L. Pelzer (E.P. 241,866, 26.8.25. Conv., 24.10.24. Addn. to 232,178; B., 1925, 907).—The method consists of a pressure distillation of the oil from a still containing a bed of filtering material such as asbestos, firebrick, kieselguhr, sand, glass wool, calcined bauxite, or finely ground petroleum coke. Oil is withdrawn from the base of the filter bed, and a portion is heated and returned to the top of the bed. Fresh oil is forced in as required, the concentration of pitch in the still contents being kept below the saturation point. It is claimed that the deposition of pitch on the still walls is by this means eliminated or very much reduced. The process is intermittent, and the filtering material may be regenerated either by burning off the adherent pitch or removing it by solvents. W. N. Hoyte.

Purification of liquids [hydrocarbon oils]. F. G. P. Remfry and A. E. Dunstan (E.P. 246,937, 13.11.24).—Mineral oils, their distillates, and the distillates of coal tar and shale oils are treated with mercuric sulphide. The process may consist in agitation of the oil with the freshly precipitated sulphide alone or in contact with some inert porous material. Treatment may be applied to the liquid or vapour phase and at any convenient temperature. The colour and odour of the oils are markedly improved. The effect is not due to chemical action. S. Bowman.

Apparatus for refining petroleum. W. H. Stilson, Assr. to Stilson Process Corp. (U.S.P. 1,564,984, 8.12.25. Appl., 20.1.20).—A still for the distillation of petroleum is connected with a horizontal vapour pipe, the temperature in which falls progressively with the distance from the still. Vertical draw-off pipes for the condensate are fitted at intervals to the underside of the vapour line, and by connecting two successive draw-off pipes together a dephlegmating by-pass is formed from which a condensate is obtained comprising material boiling between the temperatures corresponding to the distances of the pipes from the still. The outlet pipe for the condensed liquid is fitted with a trap to permit separation of any deposited carbon. T. S. Wheeler.

Conversion of hydrocarbons. H. R. Berry (U.S.P. 1,571,994, 9.2.26. Appl., 18.9.22).—Heavy liquid hydrocarbons and hot hydrogen-containing gas are caused to flow in counter current and in intimate contact under such conditions of temperature and pressure that chemical reaction takes place, the final products being lighter liquid hydrocarbons and a heavier gas containing gaseous hydrocarbons. S. Bowman.

Apparatus for separating hydrocarbons. W. C. Averill, jun. (U.S.P. 1,573,025, 16.2.26. Appl., 7.10.22).—The apparatus consists of a still connected to the lower end of a dephlegmator. Means are provided for withdrawing condensate from the lower end of the dephlegmator and introducing it again as a spray into the upper portion. W. N. Hoyte.

Condensation of hydrocarbon vapours. E. W Isom and J. E. Bell, Assrs. to Sinclair Refining Co. (U.S.P. 1,573,167, 16.2.26. Appl., 13.8.23).—The condensate obtained from the vapours from a pressure still are circulated through a cooler from and to a relatively large body of the condensate, the vapours from the still being injected into the circulating stream between the cooler and the large body of condensate. W. N. Hoyte.

Continuous treatment of natural gas gasolines. G. G. Oberfell, A. M. Ballard, R. C. Alden, E. L. Utsinger, and W. R. Lentz, Assrs. to Chestnut and Smith Corp. (U.S.P. 1,574,507, 23.2.26. Appl., 22.4.21).—The process consists in dissolving elemental sulphur in the gasoline, and passing this solution through a solution of sodium hydroxide in water. W. N. Hoyte.

Distilling oil. J. M. Wadsworth, Assr. to Pierce Petroleum Corp. (U.S.P. 1,572,584, 9.2.26. Appl., 27.12.21).—Oil is vaporised in a number of successive stages, and fractions of similar boiling point are combined and condensed to form primary distillates. These are continuously separately redistilled, the residues from the lighter distillates being added in series to the heavier distillates. H. Moore.

Transforming and distilling hydrocarbons and the like. A. A. F. M. Seigle (U.S.P. 1,572,691, 9.2.26. Appl., 16.7.20).—The space between two concentric cylindrical walls of a retort is divided by transverse partitions into a series of annular chambers. A longitudinal partition extends the length of the walls, and the transverse partitions have apertures alternately on opposite sides of the longitudinal partition. The material treated flows through each of the annular chambers in series in alternately opposite directions. Heat is applied to the inner wall of the retort. H. Moore.

Treating bituminous substances. [Desulphurising oils.] G. W. Acheson (U.S.P. 1,570,193, 19.1.26. Appl., 14.8.24).—Oil or other bituminous material is mixed with 6% of its weight of reflocculated clay prepared as described in U.S.P. 1,456,111–2 (B., 1923, 721 A; cf. also U.S.P. 1,223,350, 1,253,556, and 1,345,350–6, B., 1917, 586 A; 1918, 174 A; 1920, 564 A), 4% of concentrated sulphuric acid is added, and then a further 2% of clay. The acid transforms the sulphur present so that it is readily adsorbed by the clay, and on filtration a neutral oil containing only traces of sulphur is obtained. In place of clay other suitable adsorbents such as reflocculated carbon black may be used, and sulphuric acid may be replaced by other acid reagents, *e.g.*, chlorine. T. S. Wheeler.

Treating hydrocarbon [lubricating] oils. F. W. Hall, Assr. to Texas Co. (U.S.P. 1,570,890, 26.1.26. Appl., 20.9.21).—A process is described consisting of the upward filtration of lubricating oil through an adsorbent clay. W. N. Hoyte.

Lubricating oils. J. C. Black and J. W. Weir (U.S.P. 1,572,465, 9.2.26. Appl., 19.10.22).—Hydrocarbons are refined with sulphuric acid and the precipitated products separated. A neutralising and decolorising agent is added to the treated hydrocarbons, the mixture heated, and the hydrocarbons and admixed reagent are separated. H. Moore.

Separating wax from mineral oil distillates. S. H. Hall and C. H. Hapgood, Assrs. to De Laval Separator Co. (U.S.P. 1,571,943, 9.2.26. Appl., 8.9.21).—During the centrifuging of oil-wax mixtures, water is fed into the machine at the periphery at such a temperature that the solid wax collected there is melted and discharged with the water. S. Bowman.

Demulsification of oils etc. C. V. Zoul, Assr. to Celite Co. (U.S.P. 1,569,695, 12.1.26. Appl., 30.1.23).—An apparatus for demulsifying mixtures of petroleum hydrocarbons or the like and water comprises a tank in which demulsified oil is heated by indirect steam to 60° and dried by blowing with air. The dried oil is then circulated through a filter-press and back to the container until the temperature of the press reaches 60°. Finely divided diatomaceous earth is then added to the circulating liquid and forms a coating on the filtering material in the press. The material to be treated is passed through a heater in which its temperature is raised to 60° and is then led through the prepared filter-press in which demulsification is effected. The filtrate is run to a separating tank from which oil containing less than 1% of water is drawn off at the top and water at the bottom. In place of diatomaceous earth, wood pulp, sawdust or other granular material may be used. T. S. Wheeler.

Continuous process of demulsifying mineral oils. W. D. Leeper, Assr. to Amer. Demulsifying Co. (U.S.P. 1,573,321, 16.2.26. Appl., 7.12.21).—The oil to be treated is passed in a finely divided state through hot water and subsequently treated with live steam. W. N. Hoyte.

Apparatus for drying fuel or the like. Internat. Combustion Engineering Corp., Assees. of H. Kreisinger (E.P. 240,824, 25.9.25. Conv., 3.10.24).—See U.S.P. 1,564,361; B., 1926, 117.

Self-packing coke oven doors. A. Beckers (E.P. 248,328, 30.11.25. Conv., 25.2.25).

Construction of coke ovens. A. Beckers (E.P. 249,051, 9.12.25. Conv., 1.4.25).

Reduction of carbon dioxide to carbon monoxide (G.P. 423,945).—See VII.

Bituminous emulsions (E.P. 246,907).—See IX.

Cement from slag from gas producers etc. (G.P. 421,427).—See IX.

Treatment of waste (E.P. 247,284).—See XXIII.

III.—TAR AND TAR PRODUCTS.

Composition of coal tars. K. B. Edwards and E. S. R. Willmore (J.S.C.I., 1926, **45**, 54—56 T).—Examination of high-temperature tar by methods previously used by Edwards (*ibid.*, 1924, 143—156 T) in investigating low-temperature tar, gave neutral oils 46·6%, phenols 3·6%, rhetinols 0·9%, acids (carboxylic) 0·026%, asphalts 29·4%, ulmins 15·1%, acid ulmins 0·7%, free carbon (including ash) 3·3%. Attention is called to the similarity of the phenols from high-temperature and low-temperature tar and to the presence of large amounts of asphalts, which is considered to be indicative of high-temperature carbonisation.

Desulphurising cresols and the acid fractions from various coal tars. G. Stadnikov, N. Gavrilov, and W. Rakovski (Brennstoff-Chem., 1926, **7**, 65—68).—Crude cresols containing not more than 10—15% of neutral oils are almost completely freed from sulphur compounds of acid character (thiocresols) by treatment with formaldehyde and a condensing agent, and subsequent distillation in steam or *in vacuo*. As condensing agent a mixture of sulphonic acids from petroleum (Petroff's "Kontakt") is most effective. For example, a cresol of 2·25% sulphur content was heated for 15 hrs. in an autoclave at 120° with 1% of formalin and 1% of "Kontakt." On distilling the product *in vacuo* 85% of the cresol, with a sulphur content of 0·02%, was recovered. Equally good results were obtained when glucose syrup was used in place of the formalin. The method failed for cresols with a high content of neutral oil, and for the light oils from lignite tars. A. B. Manning.

See also A., April, 356, **System benzene–toluene** (Mitsukuri and Nakatsuchi). 381, **Hydrogenation of organic substances at high temperature under pressure** (Kling and Florentin). 389, **Cuprene tar** (Schläpfer and Stadler).

Recovery of phenols from crude ammonia liquor. Crawford.—See II.

Effect of tar and tar vapours on soil. Ewert.—See XVI.

Patent.

Purification of hydrocarbon oils (E.P. 246,937).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Electro-capillary analysis of dyes. W. Kopaczevski (Rev. Gén. Mat. Col., 1926, **30**, 34—45).—The experiments of Sahlbom have been repeated and his results verified. Electro-capillary measurements are also given for a large number of dyes,

a number of electrosols, and substances such as the albuminates of iron and copper, etc. The colloidal dyes were first purified by dialysis and the non-colloidal ones by recrystallisation, the purity being controlled by electrical conductivity determinations. Physico-chemical properties, which might affect capillarity, were determined in each case. The surface tensions and viscosities of the solutions were measured. The electric charges were determined by the method of electric transport for a fall of potential of 1·6 volts/cm. The values in doubtful cases were controlled and corrected by observations on the action of alkalis and bases on the substances studied. According to experimental results the substances worked with are classed as I. rapidly, II. distinctly, III. slowly, and IV. non-dialysable. The following conclusions are drawn: assuming the dye solutions to be not stronger than 1 in 1000 and the dyes to be in the colloidal state, acid, basic, and amphoteric dyes may be differentiated by electro-capillary analysis. The acid dyes show a rise almost equal to that of pure water, the basic hardly rise beyond the level of the solution, whilst the amphoteric dyes (indicators) are characterised by differently coloured zones. The rapidity of the rise of electro-negative and amphoteric dyes is greater than that of electro-positive dyes. The degree of dispersion has a distinct effect on the rapidity of the rise; the greater it is, the more rapid and marked the rise. Other noticeable points in electro-capillary analysis which might be of use in practical dyeing are as follows: it allows of a comparison of the rapidity of the rise of water, salts, amd colloids with reference to each other; it shows that the rise is diminished with increase in viscosity, and increased with diminution of the surface tension. Dyes which lower the surface tension of water include cochineal, Paris Violet, and Brilliant Green. Dyes which raise the viscosity of water include Trypan Red, Paris Violet, Brilliant Green, Soudan G., Direct Black W., Trypan Blue, Direct Grey 4 B, Congo Red, etc.

A. COULTHARD.

Effect of variations in concentrations of dyes in solution upon their quantitative determination spectrophotometrically. Methylene Blue. R. W. FRENCH (Ind. Eng. Chem., 1926, 18, 298—299; cf. Holmes, B., 1924, 167).—Variations within the usual limits of the concentrations of Methylene Blue used in spectrophotometric practice (2 to 10 pts. per million) are shown to have a very appreciable effect on the values of the extinction coefficient, and such effects must be taken into account in quantitative work. Values for the extinction coefficient, k, at 660 $m\mu$ have been determined for concentrations of 2, 4, 6, 8, and 10 pts. per million and found to be 2·49, 2·39, 2·30, 2·20, and 2·10 respectively. The concentrations plotted against these values of k give a straight line. As the maximum at 660 $m\mu$ increases, the slight secondary maximum at 610 $m\mu$ (cf. Holmes, *loc. cit.*) becomes progressively less. Certain oxidation products and lower homologues in the dye probably affect the maximum at 610 $m\mu$. The author agrees with Holmes that it is advisable to use alcohol as a solvent in quantitative spectrophotometric work, as in alcoholic solutions variations in absorption with changes in concentration are absent, and the maxima are more marked and easier of measurement.

A. COULTHARD.

New dye intermediates. M. T. BOGERT and R. L. EVANS (Ind. Eng. Chem., 1926, 18, 299—302).—2:4-Dinitrophenyl oxide can be prepared without risk of fire or explosion by grinding dry potassium phenoxide in excess with small quantities of chlorodinitrobenzene and slowly grinding in additional amounts of the two reactants until 400 g. of the dinitro-compound have combined with 300 g. of the phenoxide. 2-*Nitro*-4-*aminophenyl oxide* m.p., 107—108° (corr.), garnet-red prisms or orange-red plates, is obtained by the reduction of the dinitro-compound in alcohol below 60° with stannous chloride in concentrated hydrochloric acid. Reduction with alkaline sulphides is unsatisfactory. The *acetyl* derivative, m.p. 118°, pale yellow needles, is formed by warming the nitroamine with acetic anhydride. It is accompanied by diamond-shaped prisms, m.p. 124° (corr.), which also yield the original nitroamine on hydrolysis. Removal of the amino-group by diazotisation in alcoholic solution gives 2-nitrophenyl ether (cf. Haeussermann and Teichmann, A., 1918, i., 549) showing the original nitroamine to be the 2-nitro-4-amino- and not the 4-nitro-2-amino-derivative. The amine diazotises smoothly to form 4-phenoxy-3-nitrophenyldiazonium chloride, the aqueous solution of which it is possible to boil for a short time without decomposition. It couples with α-naphthol to form a rich fuchsine-red dye and with β-naphthol a brown. Phenoxy Alizarin Yellow GG, the sodium salt of which dyes silk a clear lemon-yellow, is obtained by mixing aqueous solutions of the diazotised amine, salicylic acid, and sodium carbonate at 0°. 2:4-*Diaminophenyl oxide*, m.p. 67° (corr.), colourless or pale greyish diamond-shaped crystals, is obtained by reducing the dinitro- or nitroamino-compound with iron powder and hydrochloric acid at 80—100°; the *hydrochloride* softens at about 190°, and has m.p. about 210°, the *diacetyl*-derivative has m.p. 171° (corr.). Phenoxy Bismarck Brown, blue-brown crystals, was prepared by pouring a solution of sodium nitrite into a vigorously stirred solution of the diamine hydrochloride. It dyes silk a pale orchid-brown to a dark red-brown from dilute alcohol solution. Phenoxychrysoidine, reddish-brown crystals (from diazotised aniline and the diamine hydrochloride in the presence of sodium acetate), dyes wool and silk direct in brownish-yellow to brown shades. 2:4-Dinitrophenyl sulphide, m.p. 117°, pale yellow needles, was prepared from potassium thiophenoxide and chloro-2:4-dinitrobenzene by gradually grinding the reactants together. It was reduced to 2:4-*diaminophenyl sulphide*, m.p. 107°, yellowish prisms, by means of iron and hydrochloric acid in aqueous solution under a reflux. The diaminophenyl sulphide on coupling with diazotised aniline yielded Phenylthiochrysoidine, a deep red flocculent solid, which dyes wool and silk in orange-yellow shades. The hydrochloride is crystalline.

A. COULTHARD.

Patents.

Production of colouring matter pastes [containing vat dyes]. J. Y. Johnson. From Badische Anilin- & Soda-Fabr. (E.P. 247,052, 29.5.25).—Sedimentation and the formation of a sticky deposit in vat dye pastes is retarded or prevented by the addition of soluble permanganates to the paste. For example, a concentrated solution containing 0·25 pt. or 1 pt. of potassium permanganate is added to 500 pts. of 10% Indanthrene Orange RRT paste, or of 40% Indigo RB paste, respectively.
L. A. Coles.

Ethylating amines. A. R. Cade, Assr. to Carbide and Carbon Chemicals Corp. (U.S.P. 1,570,203, 19.1.26. Appl., 17.7.23).—By the addition of an alkaline-earth hydroxide to a reacting mixture of an aromatic amine and ethyl sulphate the amine salt of ethyl hydrogen sulphate formed as an intermediate product is decomposed, thus rendering both the ethyl groups of the ethyl sulphate available for ethylation. For example, aniline (10 mols.), ethyl sulphate (11 mols.), and calcium hydroxide (12 mols.), heated under reflux at 145° for 5 hrs. give a good yield of diethylaniline calculated on both the aniline and ethyl sulphate consumed. T. S. Wheeler.

Preparation of 2-aminonaphthalene-1-carboxylic acid and its nuclear substitution products. Farbw. vorm. Meister, Lucius, & Brüning, Assees. of F. Mayer (G.P. 418,009, 19.4.23; F.P. 594,824, 2.3.25).—The products, which are of use in the manufacture of dyes and pharmaceutical products, are obtained by heating β-naphthisatin or its derivatives or substitution products with oxidising agents, preferably hydrogen peroxide, in the presence of alkalis. For example, 30% hydrogen peroxide or sodium perborate is added to a boiling aqueous solution containing β-naphthisatin and sodium hydroxide, until a red precipitate is no longer formed on acidifying a test portion of the solution with hydrochloric acid. After cooling, dilute hydrochloric acid is added to the solution until it gives an acid reaction with Congo-red, when 2-aminonaphthalene-1-carboxylic acid separates in a 90% yield. Bromo-β-naphthisatin, m.p. 297°, and dibromonaphthisatin yield respectively bromo-2-aminonaphthalene-1-carboxylic acid, m.p. 148—150°, and dibromo-2-aminonaphthalene-1-carboxylic acid. L. A. Coles.

Producing sulphur dyestuffs fast to chlorine S. Sokal. From Kalle & Co. (E.P. 248,424, 1.11.24).—See F.P. 588,874; B., 1926, 149.

Manufacture of bluish-green dyestuffs and colour lakes. O. Y. Imray. From Farbw. vorm. Meister, Lucius, & Brüning (E.P. 248,487, 9.12.24).—See F.P. 590,464; B., 1926, 149.

Preparing vat-dyestuffs. S. Sokal. From Kalle & Co. (E.P. 248,519, 30.12.24).—See F.P. 591,535; B., 1926, 149.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Use of the microscope in the textile laboratory. L. G. Lawrie (J. Soc. Dyers and Col., 1926, **42**, 73—76).—The preparation of cross-sections of textile fibres by the "celloidin" and "paraffin" methods is described. In works laboratory practice, the fibres may be simply immersed in a strong solution of celloidin, then air dried, and cut in the ordinary way. In examining dyed fibres such as those of cellulose acetate silk, organic solvents should be avoided; a mixture of gum arabic water and glycerin is recommended for binding previous to section cutting. Microscopical tests are often free from the uncertainty of chemical tests when applied to fibres, and are of particular interest in the case of artificial silks. The cross-sections of viscose and copper silks show differences which easily distinguish them. Microscopical examination is of value in investigating the nature of variations in the dyeing properties of different silks and of peculiarities in the levelling powers of dyes; in identifying the various starches used in finishing; in ascertaining the condition of vat liquors; and in investigating defects caused by mildew in fabrics etc. For counting the particles in suspension in vat dyestuff pastes the hæmacytometer may be used.
A. Coulthard.

Advances in the knowledge of fibres. R. O. Herzog (Z. angew. Chem., 1926, **39**, 297—302).—A review in which the present state of our knowledge of the structure of natural and artificial fibres is described (cf. B., 1925, 704) and an account given of the part played by the colloidal cementing substance and moisture in determining the mechanical and chemical properties of the fibre.
A. R. Powell.

Kapok and cotton: differentiation. A. Lejeune (Bull. Soc. chim. Belg., 1926, **34**, 419—421).—A method of determining the percentage of kapok and cotton in mixed fabrics is based on the facts that cotton has little affinity for dyes of the triphenylmethane group whereas kapok is readily dyed, and on the other hand that kapok is scarcely tinted by substantive cotton dyes of the benzidine group. The mixed fabric is dyed at the boil in a neutral bath with Malachite Green, and then in a slightly ammoniacal bath with Oxamine Red. Comparison with shades obtained from known mixtures of kapok and cotton enables the approximate composition of the fabric to be determined. R. Brightman.

Particular properties of the sulphuric acid esters of cellulose. A. Caille (Chim. et Ind., 1926, **15**, 189—192; cf. B., 1925, 280).—The fixation of basic dyes by cellulose derivatives is not due only to the presence of oxycellulose, but may be considerably influenced also by the presence of sulphuric acid groups. The sulphuric acid esters of cellulose, prepared under conditions excluding the formation of oxycellulose, show an affinity for basic dyes increasing proportionally with their sulphuric acid content, while their affinity for acid and direct dyes

remains unchanged. Quantitative experiments on the adsorption of Methylene Blue and Auramine show that the presence of combined sulphuric acid groups plays an important rôle in the fixation of basic dyes by nitrocelluloses, whereas the influence of the nitro-group itself is negligible. Similar phenomena are shown by the cellulose acetates, although the proportionality between dye adsorbed and sulphuric acid content is here not so apparent.

A. B. MANNING.

Utilisation of sulphite-cellulose waste liquors. S. HILPERT (Papier-Fabr., 1926, 24, 145—148).—After treatment with chlorine, sulphite-cellulose waste liquors have tanning properties. Besides chlorination, oxidation occurs with the formation of hydrochloric acid, in which the product is insoluble. The more dilute the waste liquor the further the chlorination proceeds before it is interrupted by the precipitation of the product, which, from dilute solutions, may contain 30% Cl. The higher the chlorine content the more stable the product, and the best results are obtained when chlorate and hydrochloric acid are used. A product is then obtained which dissolves more or less readily in water and tans as well as vegetable tannins. When titrated with alkali hydrochloric acid is split off, and the alkali consumption is double the equivalent of this acid. It is concluded that chlorinated quinones are present. By the action of chlorine sulphonic acid groups are also split off. Chlorinated sulphite liquor is a disinfectant; its action is strongest on *Staphylococci* and weakest on *Bac. coli*. Chloranil is similarly a disinfectant, but not its hydrolysis product, chloranilic acid. The hydrolysis of chloranil is accelerated by amino-acids and polypeptides, and it is probable that its disinfecting action is due to acid thus produced in the immediate vicinity of the bacteria.

A. GEAKE.

Carbonising sulphite [-cellulose waste] liquors with mordant salt solutions. C. G. SCHWALBE (Zellstoff u. Papier, 1926, 6, 113).—Sulphite-cellulose waste liquor, preferably previously concentrated, is treated with ferric, aluminium, or chromium sulphate, and heated for 2 hrs. at 180° and 10 atm. The organic matter is completely carbonised, and the liquor is colourless and free from organic substances. The carbon obtained contains, however, 50% of ash and is almost incombustible.

A. GEAKE.

Removal of printers' ink from old paper. E. W. ALBRECHT (Chem.-Ztg., 1926, 50, 33).—Processes previously described for the removal of printers' ink from old paper (cf. Berl and Pfanmüller, B., 1926, 000) are of theoretical interest only, as the cost is greater than the cost of production of new raw material for white paper. Old paper can be used for making dark paper without previous purification.

L. M. CLARK.

See also A., April, 353, **Elasticity of jellies of cellulose acetate** (POOLE). 387, **X-Ray investigation of cellulose and lichenin** (OTT). 441, **Sulphate content of leaf-tissue fluids of Egyptian and Upland cotton** (HARRIS, HOFFMAN, and HOFFMAN).

Dry cleaner's naphtha. JACKSON.—See II.

Influence of soluble silicates on hypochlorite bleach. CARTER.—See VI.

Arsenic in burner gases and its bearing on "Haff" disease. GLASER.—See VII.

PATENTS.

Deodorising hair, feathers, and the like. Treating apparatus. A. H. GALLAGHER, Assr. to TANNERS PRODUCTS Co. (U.S.P. 1,574,617–8, 23.2.26. Appl., 24.5.24).—(A) Animal tissues, such as hair, feathers, and the like, are deodorised by treatment with ozone. Intimate contact with the ozone is obtained by keeping the material in motion, and the resilient quality of the material is retained. (B) Apparatus for this treatment consists of a chamber into which the material is delivered in a current of gas. Means are provided for circulating the material within the chamber and for introducing ozone.

A. GEAKE.

Liquid treatment and subsequent pressing of pulp. J. BRANDWOOD (E.P. 247,307, 16.8.24). Wood pulp is impregnated with a liquid, *e.g.*, an alkali in the preparation of viscose, and the excess liquid removed in a continuous process. The pulp travels on an endless band conveyor successively through a tank containing the liquid, and a press. During its passage through the tank the pulp is held on the conveyor by a second endless band above it. The upper plate of the press is lowered at regular intervals, and during its downward travel the motion of the conveyor automatically ceases.

A. GEAKE.

Cooking wood chips. J. T. JAEGER (U.S.P. 1,572,723, 9.2.26. Appl., 1.4.22).—The vessel containing the chips is provided with a steam supply pipe, an acid conveying pipe, and an acid circulating pipe leading from one end and discharging into the opposite end of the vessel through a nozzle. The discharge nozzle consists of a length of pipe flared at the front end and covered with a perforated head which is fixed with regard to the flared end so that acid can be discharged through the side openings as well as through the perforations.

B. W. CLARKE.

Preventing froth on paper machines. J. A. DE CEW, Assr. to PROCESS ENGINEERS, INC. (U.S.P. 1,573,814, 23.2.26. Appl., 14.12.21).—Frothing of paper pulp sized with rosin and alum is prevented by adding a soluble iron salt, whereby the soluble substances which cause the frothing are precipitated.

A. GEAKE.

Conservation of pellicles of hydrate or of hydrated ethers of cellulose. J. DEFAUCAMBERGE, Assr. to SOC. FRANÇ. DES CRINS ARTIFICIELS (U.S.P. 1,576,631, 16.3.26. Appl., 12.1.24).—See E.P. 224,487; B., 1925, 126.

Regenerating sulphurous acid and waste heat from sulphite-cellulose boilers. C.

Hangleiter and A. Schneider, Assrs. to Zellstofffabrik Waldhof (U.S.P. 1,576,970, 16.3.26. Appl., 3.4.22).—See G.P. 350,471; B., 1922, 855 a.

Humidifying and impregnating textile and other materials. A. Manzoni and E. Muller (E.P. 243,359, 18.11.25. Conv., 18.11.24).

Apparatus for the manufacture of artificial filaments. L. A. Levy (E.P. 248,468, 4.12.24).

Decolorising carbon (E.P. 246,954).—See II.

Treatment of waste (E.P. 247,284).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Soluble silicates; their influence on hypochlorite bleach. J. D. Carter (Ind. Eng. Chem., 1926, 18, 248—252).—The addition of sodium silicate neither hastens nor retards the loss of available chlorine from hypochlorite solutions alone, but in the presence of a bleachable material the rate of loss is much reduced. The loss in strength of cheesecloth after bleaching with hypochlorite is reduced by the use of silicate, as is shown by determinations of bursting strength. The ash content of the cloth is considerably increased by silicate treatment, silica being deposited in the fabric. It is also claimed that the cloth is rendered brighter and whiter. In the bleaching of wood pulp the use of sodium silicate has no effect if added to sodium hypochlorite; with calcium hypochlorite, however, improved bleaching results, apparently an effect due to a flocculent precipitate of calcium silicate. The most alkaline sodium silicate of commerce gives the best results in this connexion, and it is recommended to add a quantity equal to 1% of the weight of air-dry pulp after the bath has been made up. C. Irwin.

Weighting of silk. R. O. Herzog and H. W. Gonell (Z. angew. Chem., 1926, 39, 380—381).—The *X*-ray examination of silk fibres weighted with various materials, such as tin phosphate and water-glass, shows interference rings superimposed upon the original point pattern of the silk itself, except in the case of alumina treatment, where the silk diagram is obscured owing to separation of amorphous material. This shows that there is no chemical combination between the weighting material and the silk substance itself; probably the weighting material, which takes a crystalline form, penetrates into the inter-cellular spaces of the fibre and is fixed by the degradation products of the fibrous material.
B. W. Clarke.

Electrocapillary analysis of dyes. Kopaczevski.—See IV.

Sulphuric acid esters of cellulose. Caille.—See V.

Patents.

Bleaching textiles by means of ozone. E. Crespi and M. P. Otto (E.P. 247,738, 16.2.25).—Textile material is treated with weak acids, dried, and bleached by treatment with a current of moist, ozonised air at the ordinary temperature and pressure, after which it is preferably washed with water. Apparatus for the purpose is described.
L. A. Coles.

Dyeing and printing of textile materials. E. Knecht and E. F. Muller (E.P. 247,694, 16.12.24).—The fastness to washing of dyeings with direct cotton dyes is improved by dyeing with water-soluble zinc or magnesium compounds of the dyes, in the presence of ammonium salts together with ammonia or sodium bicarbonate in quantity insufficient to precipitate basic compounds. Zinc or magnesium compounds of the dyes are deposited in the fibres, where they are subsequently fixed by heating or steaming. Ammonia or sodium bicarbonate need not be added if ammonium salts of volatile acids, such as ammonium acetate, are used. For example, 100 lb. of cloth are dyed in a 200-gal. bath containing 3 lb. of dye, 75 lb. of magnesium sulphate, 18 lb. of ammonium sulphate, and 5 gal. of ammonia, *d* 0·880, the ammonia being added 15 min. after the other constituents of the bath. Compositions containing the water-soluble zinc or magnesium compounds may be used for printing.
L. A. Coles.

Resists for use in dyeing and printing. S. W. Clarke and J. R. Brown (E.P. 247,757, 11.3.25).—Liquid or paste for use as a resist, is prepared by stirring 2 pts. of flour into a mixture of 1 pt. of gum arabic and 2 pts. of glue in which the glue has previously been softened by heating with a suitable quantity of water. The resist is coated on to portions of the fabric and allowed to dry before dyeing, or, in the case of hanks, string or tape soaked in the resist is wound around portions of the hank to be protected from the dye liquor, or between two portions of the hank which are to be dyed in different baths. The resist is subsequently removed from the material by washing. L. A. Coles.

Manufacture of soluble vat dyestuff preparations. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 248,811, 8.11.24).—See F.P. 589,914; B., 1926, 154.

Dyeing, washing and like apparatus. British-Amer. Laundry Machinery Co., Ltd. From Amer. Laundry Machinery Co. (E.P. 248,793, 8.9.24).

Yarn dyeing apparatus. W. J. Mellersh-Jackson. From Fuld & Hatch Knitting Co. (E.P. 249,369, 27.5.25).

Apparatus for dyeing or otherwise treating yarns and other materials. T. McConnell (E.P. 249,406, 9.9.25).

Machines for dyeing, scouring and washing wool and other fibrous materials. J. and R. Whitaker (E.P. 249,413, 21.9.25).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Presence of arsenic in burner gases and its bearing on "Haff disease." F. GLASER (Chem.-Ztg., 1926, 50, 185; cf. Biginelli, B., 1926, 302).—The presence of sodium chloride in pyrites used for the production of sulphur dioxide results in the formation of minute amounts of hydrogen chloride in the roasting operation; when these gases come into contact with strong sulphuric acid volatile arsenic trichloride is formed from any arsenious acid present and this is the probable cause of the poisoning of platinum contact masses. When the sulphur dioxide is used for the preparation of cellulose, it is probable that some of the arsenic trichloride or other volatile arsenic compound formed in the process escapes into the air or in the effluent waters in which, if they are salt as is the case at the Königsberg Haff, they are more stable than in fresh water. The presence of this arsenic in the air gives rise to the so-called "Haff disease."

A. R. POWELL.

Production of hydrogen chloride from chlorine and steam in the presence of carbon. B. NEUMANN and R. DOMKE (Z. angew. Chem., 1926, 39, 368—374).—Chlorine reacts quantitatively with water vapour in the presence of wood charcoal above 600° with the formation of hydrogen chloride, an excess of steam being necessary to complete the reaction. The temperature for quantitative reaction is 450° with coke and 350° with active charcoal. The increased reactivity in the presence of coke is due to the ferric oxide in the coke; a mixture of wood charcoal and ferric oxide brings about the reaction at 350°. At 450° complete interaction is obtained after a contact of only 3 sec. The hydrochloric acid produced by cooling the gaseous products has a concentration of 32—35%; it is free from chlorine, arsenic, and, with the exception of the case of coke, from sulphur. A proportion of the chlorine is adsorbed by the carbon during the process and cannot be removed by steaming. The primary reaction is probably: $2H_2O+2Cl_2+C=4HCl+CO_2$. The carbon dioxide is then reduced to some extent by more carbon to form carbon monoxide, which is always present in the gaseous products. B. W. CLARKE.

Production of hydrogen bromide from bromine and water vapour in the presence of carbon. B. NEUMANN, W. STEUER, and R. DOMKE (Z. angew. Chem., 1926, 39, 374—375).—The reaction between bromine and water vapour is complete at 600° in the presence of wood charcoal, and at 500° if ferric oxide is mixed with the charcoal. The concentration of the hydrobromic acid produced depends on the excess of steam employed in the process, and acid up to 60% (fuming hydrobromic acid) can be obtained. The concentrated acid is slowly decomposed on exposure to the air or by the action of light with the formation of free bromine. The use of this process avoids the formation of acid mists which are difficult to condense. B. W. CLARKE.

Factors affecting the fixation of nitrogen as sodium cyanide. E. W. GUERNSEY, J. Y. YEE, J. M. BRAHAM, and M. S. SHERMAN (Ind. Eng. Chem., 1926, 18, 243—248; cf. B., 1925, 669).—The equilibrium conditions of the reaction $Na_2CO_3+N_2+4C+Fe=2NaCN+3CO+Fe—138{,}500$ cal. were investigated on a laboratory scale. Under most conditions the conversion-time curve shows a maximum, the percentage conversion then decreasing. Silica introduced accidentally with the iron catalyst had a deleterious effect, 1 mol. SiO_2 reducing 2 mols. Na_2O and removing it from the desired reaction. The reaction being reversible, increase in the rate of nitrogen supply reduces the proportion of carbon monoxide in the gas and increases the conversion. Rise of temperature has the same effect and if only a reasonable excess of nitrogen is to be used a temperature not below 1050° is desirable. Under these conditions 1—2% of iron in the charge would be sufficient. The reactivity of metallurgical coke is only a small fraction of that of petroleum coke, wood charcoal, or lampblack. In all cases a portion of the charge sublimed and was recovered as a deposit consisting mainly of sodium carbonate. Too long treatment, high gas velocities, or the use of inactive coke or catalyst resulted in the occurrence of metallic sodium in the sublimate. It is concluded that sodium cyanide is partly dissociated in the vapour phase and cannot be recovered by distillation.

C. IRWIN.

Titration of ammonium sulphate by the stearate method. H. ATKINSON (Analyst, 1926, 51, 140—141; cf. A., 1926, 38).—The method previously described was found applicable to the determination of ammonium sulphate. Ten c.c. of a solution of ammonium sulphate approximately 0·1*N* strength were diluted to 40 c.c., and the indicator and 0·5 c.c. of stearate solution added. Excess of 0·1*N*-barium chloride solution was then added and the excess titrated with stearate solution. One or two drops of barium chloride changed the green colour to lemon-yellow, and this solution was used for comparison in the titration of solutions containing known quantities of the ammonium sulphate solution. The correction to be added to the titration results was thus found to be 0·22 for 10 c.c. of ammonium sulphate solution, and the error of the method was found to be less than 1 drop in 10 c.c. of solution. Weak acids such as hydrogen sulphide and carbonic acid must be removed by boiling the acid solution.

D. G. HEWER.

Determination of the acidity of commercial ammonium sulphate. H. M. LOWE (Gas World, 1926, 84, Coking Sect., 30—31).—The use of methyl-orange as an indicator in the titration of ammonium sulphate is discussed. It is suggested that acidity in this salt should be determined by matching the colour with methyl-orange against a range of standard solutions of the pure salt with known amounts of acid added. C. IRWIN.

Neutrality of ammonium sulphate. T. B. SMITH (Gas World, 1926, 84, Coking Sect., 26—28).

—The determination of free acid in ammonium sulphate by titration, using methyl-orange as indicator, gives results indicating a higher degree of acidity than the electrometric method. The error in the former method is due to the presence of pyridine sulphate which is acid to methyl-orange. A closer approximation to accuracy is given by the following procedure. Two 50-g. lots of the sample are weighed. One is mixed with excess of ammonia (*d* 0·88) and dried below 60°. Both lots are dissolved in an equal volume of water, filtered, and the unneutralised sample titrated with 0·1*N*-sodium hydroxide solution until the colour with methyl-orange matches that of the neutralised one.

C. IRWIN.

Caking of ammonium sulphate. G. J. GREENFIELD (Gas World, 1926, 84, Coking Sect., 28—29).—The cause of caking is ultimately the alternate taking up and loss of atmospheric moisture by the salt. The crystal size is, however, important. A coarse salt offers less surface than a fine one, but if fines are present along with coarse crystals they fill the interspaces and promote caking. An even crystal size is desirable and hence processes which aim at allowing the crystals to grow in the saturator are of no advantage as the crop is irregular. The use of ammonium carbonate in place of sodium carbonate as neutralising agent gives a salt which dries more easily and with less breaking up of crystals. In all cases the crystals should be cooled immediately after drying and stored under constant atmospheric conditions.

C. IRWIN.

Analysis of silicates. O. CANTONI (Annali Chim. Appl., 1926, 16, 92—95).—In the evaporation of acid solutions containing silicic acid, to render it insoluble, the presence of the chlorides of iron, aluminium, calcium, and magnesium often causes the formation of tenacious pellicles, so that several days may be required to evaporate to complete dryness on the water bath. The addition of a few grams of sodium chloride (or other non-hygroscopic salt) is stated greatly to shorten this time of evaporation. The salt may be easily eliminated subsequently by washing. To render the hydroxides of the metals of the third group more easily filterable, it has been suggested to precipitate them with the aid of filter paper pulp, but this procedure may cause incomplete precipitation. Washed kaolin, of which the exact loss on heating to constant weight is known, is more satisfactory; it should be added immediately after precipitation by ammonia, with strong agitation. In the fusion of iron and aluminium oxides with potassium bisulphate, the time required may be reduced from 24 hrs. to ½ hr. by increasing the proportion of bisulphate from the usual 10 pts. to 20 or 30 pts. A method which shortens the time of separation of the alkalis is also given (cf. B., 1925, 831), depending on the nearly complete removal of the other metals by heating the residue obtained after the removal of silicic acid with hydrofluoric acid to a low red heat, so as to drive off most of the sulphuric acid.

S. B. TALLANTYRE.

See also A., April, 331, **Mesothorium-2** (YOVANOVITCH). 332, **Chemical properties of mesothorium-2 and actinium** (GLEDITSCH and CHAMIE); **Highly emanating radium preparations** (HAHN and HEIDENHAIN). 340, **Melting points of barium, strontium, and calcium oxides** (SCHUMACHER). 345, **Solubility of double nitrates of praseodymium and neodymium with bivalent metals** (PRANDTL and DUCRUE); **Influence of citrates on precipitation of barium sulphate** (NICHOLS and THIES, JUN.). 358, **Phase equilibria of sulphates** (JÄNECKE); **Equilibrium in system arsenic pentoxide–barium oxide–water** (HENDRICKS). 359, **Thermal dissociation of metal sulphates** (MARCHAL); **Explosion range of hydrogen–ammonia–air and hydrogen–ammonia–oxygen mixtures** (JORISSEN and ONGKIEHONG). 362. **Oxidation of ammonia to nitrite in aqueous solution** (MÜLLER). 363, **Velocity of hydrolysis of aqueous solutions of alkali cyanides** (ZAWIDZKI and WITKOWSKI); **Reaction between sodium sulphite and sulphur** (WATSON and RAJAGOPALAN).

Detecting small quantities of oxygen in gas mixtures. SCHULEK.—See XXIII.

PATENTS.

Manufacture of nitric acid from the nitrous gases arising during the catalytic combustion of mixtures of ammonia and oxygen. I. W. CEDERBERG (E.P. 246,889, 1.10.24).—A mixture of ammonia with the least possible excess of oxygen over that required by the equation, $NH_3+2O_2=H_2O+HNO_3$, is passed through a catalyst chamber under atmospheric pressure, or under just sufficient excess pressure to ensure the flow of gases through the chamber. The products subsequently pass through absorption apparatus, *e.g.*, U-tubes constructed of ferrosilicon, or towers filled with acid-resisting material, in which the gases and condensed liquid flow in the same direction, no water being added to the gases. Nitric acid of about 70% strength is obtained.

L. A. COLES.

Lead chambers employed in the manufacture of sulphuric acid. W. G. MILLS, and PACKARDS AND JAMES FISON (THETFORD), LTD. (E.P. 247,644, 17.11.24).—A lead chamber of circular cross-section, in which the gas inlet is tangential to the chamber wall and comparatively near the bottom. The outlet is still nearer the bottom on the other side of the chamber.

C. IRWIN.

Recovering sulphur dioxide [from smelting furnace gases]. A. H. EUSTIS (Can. P. 248,607, 2.5.24).—Sulphur dioxide is absorbed either in a liquid or in a solution of some substance with which it will form an additive compound, and the partial pressure of the sulphur dioxide above the liquid is then reduced by introducing a large quantity of some other gas, so liberating the sulphur dioxide. This may be done by running the solution into another chamber and blowing in exhaust steam.

E. S. KREIS.

Purification of hydrocyanic acid [from hydrogen sulphide]. M. Walker, Assr. to Pacific R. and H. Chemical Corp. (U.S.P. 1,571,982, 9.2.26. Appl., 26.10.25).—Hydrogen sulphide present as an impurity in liquid hydrocyanic acid is removed by treatment with sulphur dioxide. L. A. Coles.

Manufacture of iron perchloride. D. Tyrer (E.P. 248,214, 6.5.25).—Ferric chloride is obtained by chlorinating crystalline ferrous chloride, and adding sufficient water to yield crystalline ferric chloride, or a solution of desired strength. L. A. Coles.

Manufacture of alkali phosphates. C. M. Hoff, Assr. to Grasselli Chemical Co. (U.S.P. 1,572,846, 9.2.26. Appl., 17.7.24).—Alkali phosphate solution is electrolysed in a diaphragm cell, and alkali phosphates are recovered separately from the anode and cathode solutions. L. A. Coles.

Separation and recovery of arsenic and/or antimony and tin. J. A. Lahey, Assr. to Vulcan Detinning Co. (U.S.P. 1,575,217, 2.3.26. Appl., 6.5.25).—A solution containing the arsenic and tin in the form of arsenate and stannate is evaporated and cooled to allow the arsenic salt to crystallise. Tin is recovered from the filtrate. A. R. Powell.

[Preparation of] silicofluorides and fluorides. Chem. Fabr. Griesheim-Elektron (F.P. 599,278, 9.6.25. Conv., 10.6.24).—Calcium fluoride is mixed with a salt of an alkali or alkaline-earth metal, the silicofluoride of which is of slight solubility, and also with silica and with an acid forming a soluble calcium salt. The silicofluoride produced is converted by alkali compounds into alkali fluoride. C. Irwin.

Decomposition of reducible substances [phosphates]. G. M. Lombardo (F.P. 599,285, 9.6.25).—Natural phosphates or the like are heated with carbon in absence of air and the volatile decomposition products are burnt by blowing with air in the proximity of the zone of reaction, their heat of combustion thus being utilised. C. Irwin.

Preparation of thenardite from sodium chloride and magnesium sulphate. Kali-Forschungs-Anstalt G.m.b.H. (G.P. 424,083, 21.4.21).—The double sulphate of sodium and magnesium is decomposed into Glauber's salt and anhydrous magnesium sulphate, and the former dehydrated with sodium chloride at a definite temperature. C. Irwin.

Preparation of pure aluminium sulphate. Chem. Fabr. Griesheim-Elektron (G.P. 424,129, 20.4.19).—Pure crystalline aluminium chloride prepared by the action of hydrochloric acid on alumina is decomposed at a temperature of 115° with concentrated sulphuric acid, a great excess of this being avoided. C. Irwin.

Preparation of concentrated colloidal arsenious sulphide solution. O. Faust (G.P. 424,141, 11.1.25).—Hydrogen sulphide is passed into a solution of arsenious oxide in pure or aqueous glycerin, preferably in presence of a protective colloid. C. Irwin.

Manufacture of zinc oxide. N. V. Handelmaatschappij Grikro (E.P. 225,833, 7.11.24. Conv., 3.12.23).—In the process of vaporising zinc and burning the vapour, condensation and oxidation of zinc fumes around the outlet-hole of the vaporising chamber cause the formation of accretions which obstruct the flow and reduce the yield of zinc oxide unless continually stripped off. To avoid this a current of "protective" gas, *e.g.*, producer gas, or coke-oven gas (preferably preheated), is directed against the edges of the fume outlet. S. S. Woolf.

Manufacture of zinc oxide. H. H. Utley and J. McCallum, Assrs. to River Smelting and Refining Co. (U.S.P. 1,574,317, 23.2.26. Appl., 7.6.23).—A fume product containing zinc and lead is treated with chlorine, the lead chloride removed by volatilisation, and the residue roasted with carbonaceous reducing material. C. Irwin.

Production of carbon disulphide from ammonium thiocyanate. J. A. du Bois (G.P. 423,860, 23.11.23).—Carbon disulphide is produced by heating ammonium thiocyanate with steam, *e.g.*, by dropping a solution of the salt into a heated vessel fitted with a reflux condenser to condense undecomposed ammonium thiocyanate. The uncondensed vapours are passed through hot sulphuric acid. L. A. Coles.

Apparatus for continuous distillation of sulphur. Raffineries Internat. de Soufre (F.P. 598,407, 5.9.24).—The apparatus consists of a melting pot, storage tank, and distillation chamber, connected in such a way that the level of sulphur in the distillation chamber is kept constant without the use of mechanical regulating devices. The flue-gases are used to melt the sulphur and also, playing around the feed pipe connecting the storage tank to the distillation chamber, raise the temperature of the feed sulphur to that of the sulphur in the distillation chamber. E. S. Kreis.

Mechanical salt-cake furnaces. Farbw. vorm. Meister, Lucius, & Brüning (E.P. 227,799, 27.11.24. Conv. 18.1.24. Addn. to 225,548).—See G.P. 414,971; B., 1925, 848.

Production of potassium salts. E. Niccoli (E.P. 247,405, 6.3.25).—See F.P. 594,904; B., 1926, 237.

Adsorbent materials (U.S.P. 1,570,537).—See I.

Distillation of coal (E.P. 243,665).—See II.

Hydrogen (F.P. 599,614).—See II.

VIII.—GLASS; CERAMICS.

Manufacture of transparent silica glass. H. George (Compt. rend., 1926, 182, 850—851).—Transparent silica, free from gas bubbles, is obtained by fusion of certain very pure natural quartzites which are built up of microscopic crystals of silica and contain no occluded gases. This quartz does not crack or disrupt on heating to fusion. S. K. Tweedy.

Ultramicroscopic motion picture study of the relation of colloidal content and plasticity in clays. W. G. France (J. Amer. Ceram. Soc., 1926, 9, 67—76).—The colloidal fractions of four clays were studied by the aid of the kinematographic camera and the ultramicroscope, the number and extent of the light areas on the photographic prints being taken as an indication of the quantity of colloidal matter present. The results obtained indicated a definite relationship between the colloidal content and the plasticity of the clays examined. Arranged according to decreasing plasticity (*i.e.*, colloidal content) the order of the four clays was: English china clay, S. Carolina kaolin, N. Carolina kaolin, fireclay. The effect of a deflocculating agent (sodium hydroxide) and of a flocculating agent (acetic acid) was also observed by this method.
F. Salt.

Minerals of bentonite and related clays and their physical properties. C. S. Ross and E. V. Shannon (J. Amer. Ceram. Soc., 1926, 9, 77—96).—The main object of the work was to determine whether the minerals of the bentonite group of clays could be studied and explained by ordinary mineralogical methods. Bentonite is defined as a rock composed essentially of a crystalline mineral formed by devitrification of a glassy, igneous material, usually a tuff or volcanic ash. The characteristic clay-like mineral is usually montmorillonite, less frequently beidellite; it has a micaceous habit, facile cleavage, and high birefringence. The structure of the original rock is retained to a remarkable degree in the clay. The high adsorptive powers of bentonite are due to its micaceous structure and easy cleavage, giving great surface area, and to its felt-like texture, and not to the presence of colloids; it contains no gel colloids and few of its crystalline particles can be included within colloidal dimensions. Chemical analyses of purified bentonites showed them to consist essentially of a mineral closely resembling the French montmorillonite. The amount of bivalent bases (magnesia and lime) is approximately constant; they are therefore assumed to form an essential part of the molecule. Hence, the suggested formula is: $R''O$, R'''_2O_3,$5SiO_2$,5—$7H_2O$. The water content varies from 16·10% to 25·67%, according to the temperature and humidity of the atmosphere. A large proportion is driven off at 120°, only 5% or less being retained above 450°. One clay was found, on analysis, to consist largely of beidellite, having the formula Al_2O_3,$3SiO_2$,$4H_2O$. Another, completely crystalline and micaceous, had a composition corresponding to halloysite, Al_2O_3,$2SiO_2$,nH_2O. The optical properties of the clay minerals are discussed. F. Salt.

Elimination of limestone from clays. R. A. Horning (J. Amer. Ceram. Soc., 1926, 9, 110—113).—After the removal of the larger stones by hand, the clay, with the smaller stones, is fed to a pair of conical rollers, one threaded, the other smooth, spaced about ¼ in. apart. All stones larger than one inch are thrown out by the threaded roller; smaller stones are crushed to pea-size or smaller. Finally, the clay, after being mixed with water and pugged, is passed through a set of smooth rollers, 18 in. in diameter, set less than 1/16 in. apart. The wet, smooth stones are not readily gripped by these rollers, and are removed by means of an oscillating finger, whilst the smallest stones are crushed to powder, which is intimately mixed with the clay in a final pugging operation. F. Salt.

Influence of ferric oxide and titanium oxide on pure clays. A. H. Kuechler (J. Amer. Ceram. Soc., 1926, 9, 104—109).—The effect of small additions of titania and of ferric oxide on the fusion temperature of kaolin and of fireclay was investigated. The binary graph showing the effect of additions of ferric oxide tends towards a straight line; the irregularities are accounted for by the fact that ferrous iron is a more powerful flux than ferric iron. Approximately 10% Fe_2O_3 lowers the fusion temperature of kaolin and fireclay by 6 and 6½ cones respectively. The corresponding graph for titania is a straight line in the case of kaolin, but slightly irregular in the case of fireclay, owing to the iron content. About 5% TiO_2 lowers the fusion temperatures by 2 and 3 cones respectively. The study did not reveal any eutectic. F. Salt.

Influence of composition of body and glaze on the physical properties of a true porcelain. P. W. Lee (J. Amer. Ceram. Soc., 1926, 9, 97—103).—An attempt was made to develop a porcelain of high quality mainly from American raw materials; the only imported material was English china clay. The results are presented in three-phase diagrams. The best body mixture produced contained 47·5% of clay, 28·9% of flint, 22·7% of felspar, 0·6% of calcium carbonate, 0·2% of magnesium carbonate, and 0·1% of boric acid. The best colour for each body was produced at a definite temperature; the total content of alkaline-earth fluxes must not exceed 1%, or that of magnesium carbonate, 0·4%. The zinc content of the glaze improved the white colour, but decreased the transluceney and produced a rough glaze. F. Salt.

Recent researches on porcelain. E. E. Urbschat (Z. angew. Chem., 1926, 39, 365—368).—The author gives a brief review of recent researches on the m.p. diagram for alumina–silica mixtures, in relation to the firing of clays and clay products; on the variations in physical (including optical) and mechanical properties of porcelain made with various types of quartz; on colours and glazes for ceramic products; and on the factors influencing the life and durability of the saggers for firing porcelain. B. W. Clarke.

Monograph and bibliography on terra cotta. H. Wilson (Bull. Amer. Ceram. Soc., 1926, 5, 94—145).—Summarised information is given on terra cotta, its uses, durability, methods of manufacture, physical properties, etc. A full bibliography gives 173 references, mainly to American literature.
F. Salt.

Silica bricks. Dilatometric analysis of raw materials. Travers and de Goloubinoff (Rev.

Mét., 1926, 23, 100—117 ; cf. B., 1926, 274).—A review of the various methods used to identify the allotropic forms of silica occurring in silica bricks. The usual method for determining the absolute density by means of a specific gravity bottle gives low results due to entrapped air and this error is increased with increased fineness of grinding. The use of petrol in place of water showed a complete elimination of air bubbles after shaking with the powder for a few minutes only at atmospheric temperature. The expansion curve of cristobalite showed a break at 210°, and that of tridymite at 440—450°. Above 450° the curve for the expansion of tridymite up to 1100° is a straight line with a very small value for the coefficient of expansion. The total expansion between 440° and 575° is given as quartz 0·77%, cristobalite 0·055%, tridymite 0·06%. In commercial bricks containing a mixture of the allotropic forms of silica, determinations by dilatometric methods enable cristobalite (35—70%) to be estimated with an error of 2—3%, and quartz (10—30%) and tridymite (25—30%) within 2—5%. The sum of the constituents thus found is generally from 80 to 85% and the difference to 100 is explained by the presence of fused impurities and also small quantities of amorphous silica. Contrary to the conclusions of Endell (cf. B., 1913, 426) β-cristobalite was not obtained by chilling from above the transition point of the α-form. C. A. KING.

Coagulation of clay. GALLAY.—See A., April, 354.

PATENTS.

Vitreous composition [resistant glass]. E. C. BUCK (U.S.P. 1,570,202, 19.1.26. Appl., 26.2.24).—A glass comprising a complex metaphosphate of aluminium and an alkaline-earth metal, and having the composition, P_2O_5 73—78%, CaO or MgO 23—10%, and Al_2O_3 3—13%, and substantially free from silica, is resistant to the action of hydrofluoric, hydrofluosilicic, and phosphoric acids, and can be readily worked. Such a glass containing lime has a relatively high coefficient of thermal expansion which falls as the lime is replaced by magnesia ; by using suitable proportions of lime and magnesia a glass suitable for lining silica glass vessels can be prepared. For manufacture calcium hydrogen phosphate, aluminium phosphate, and phosphorus pentoxide are fused together in suitable proportions, silica being removed by addition of a small quantity of aluminium or calcium fluoride or by using an excess of phosphorus pentoxide. T. S. WHEELER.

Drying of clay products and other goods. A. E. BROWN (E.P. 246,935, 12.11.24).—The goods to be dried, which are kept stationary throughout the process, are arranged in a series of chambers provided with longitudinal flues above the ceiling and below the floor. Hot air is admitted from above and distributed evenly through the chamber by openings in the flue arranged to deliver approximately equal amounts of air. The air, slightly cooled and containing some moisture, is withdrawn from the lower flue, after a relatively slow vertical passage through the chamber, and the air from the last of the series of chambers is passed in increasing quantities (to compensate for its lower heat content) through the chambers containing goods in a wetter state, *i.e.*, goods liable to be damaged by rapid drying. B. W. CLARKE.

[Brick] kilns. STONEWARE, LTD., and H. DEAN (E.P. 247,245, 13.6.25).—In kilns adapted for drying and burning bricks, tiles, etc. (particularly blue bricks), there are two groups of chambers, built in parallel and having a space between the groups to afford access to the chambers. The chambers are internally fired, and are interconnected by means of damper-controlled apertures in the partition walls ; they may be heated in series from a fire in one. A pair of interconnected chimney flues extends along the outer sides of the two parallel groups and each flue is connected by damper-controlled connexions with each chamber of its group. The two branches of the chimney flue are provided with dampers so that the flow of gases in the chambers may be reversed. Connecting ducts controlled by dampers are provided between each chamber and the flues, and by this means any of the chambers in either group may be put into communication with any other chamber and the heat extracted from one chamber or group of chambers may be utilised for the drying of goods in other chambers prior to burning. A. W. HOTHERSALL.

Brick-making. H. M. RIDGE (E.P. 247,325, 28.11.24).—In the kiln-burning of bricks, a portion of the evolved hydrocarbon gases is drawn off at a part of the kiln which is sufficiently hot to prevent condensation on the bricks. The whole or a part of the gases so drawn off is returned to the combustion zone of the kiln where it is burnt with the aid of added air. Where there is an excess of hydrocarbons over that required to heat the kiln, it may be utilised for other purposes, or it may be cooled in order to condense part or all of the hydrocarbons. A. W. HOTHERSALL.

Drying refractory materials. W. E. WILSON and H. G. LYKKEN (U.S.P. 1,570,659, 26.1.26. Appl., 11.12.23).—The ware is subjected to rising temperature and decreasing humidity in passing through a heating and humidifying medium, which circulates in a general direction opposite to that in which the ware travels. At successive points the drying medium is forced in a vertical direction between the pieces of ware. F. SALT.

Dryer kiln [for ceramic ware]. H. R. STRAIGHT (U.S.P. 1,572,326, 9.2.26. Appl., 7.2.24).—A drying tunnel is provided with a series of upper and lower compartments through which air is circulated. The roof of each upper compartment is formed of a horizontal portion and a curved portion which approaches asymptotically to the line of travel of the ware being dried, so that the air is reflected downwards through the ware. The lower compartments are staggered with regard to the upper, and the floor is formed horizontally with a curved portion

which approaches asymptotically to the line of travel of the ware towards the delivery end and directs the air upwards through the ware.
B. W. Clarke.

Continuous kiln. H. R. Straight (U.S.P. 1,572,327, 9.2.26. Appl., 17.3.24).—The ware is moved on cars through a tunnel designed so that the space between the cars and the walls of the tunnel may be sealed, dividing the tunnel into two compartments above and below the floor line of the cars. Both ends of the lower compartment and the inlet end of the upper compartment may be sealed, and the upper compartment is heated by the products of combustion of a furnace. The air necessary to maintain combustion is drawn mechanically through the lower compartment. B. W. Clarke.

Batch for making ceramic material. F. M. and F. J. Locke (U.S.P. 1,572,730, 9.2.26. Appl., 17.10.24).—The raw batch for making a refractory material is made up of a relatively small amount of a compound of manganese and a relatively large amount of clay. B. W. Clarke.

Manufacture of refractory material. Ateliers de Construction Oerlikon (F.P. 596,461, 8.4.25. Conv., 22.5.24).—Mixtures of grog, graphite, and clay for the manufacture of crucibles, muffles, and the like are moistened with a solution containing borax and boric acid in the proportions $Na_2B_4O_7$ to $4H_3BO_3$.
B. W. Clarke.

Forming sheet glass. E. C. R. Marks. From Libbey-Owens Sheet Glass Co. (E.P. 248,943, 8.4.25).

IX.—BUILDING MATERIALS.

Refractory hydraulic cement. J. Arnould (Chim. et Ind., 1926, 15, 184—188).—Attempts have been made to prepare a cement with good setting properties, and capable of withstanding high temperatures without deterioration, using mixtures based respectively on Portland cement, magnesia cements, and aluminous cements. The last alone proved suitable as a basis for a refractory cement, the mixture most nearly satisfying the requirements consisting of 1 pt. of aluminous cement (*ciment fondu*) and 2 pts. of calcined and powdered bauxite. This gives a cement which sets slowly and hardens rapidly, and undergoes only slight shrinkage on heating. It softens, however, at 1350—1400° and melts at about 1600°. Mixed with crushed refractory material in the proportion of 1—3 pts. of crushed refractory to 3 pts. of cement, it gives a concrete which is not affected by rapid changes of temperature. After heating to 1000° the cement and concrete exhibit a certain degree of friability, but this disappears again on heating to 1250°. The manufacture of this cement (*ciment Kestner*) is now being undertaken in France.
A. B. Manning.

Patents.

Impregnating wood. Gebr. Himmelsbach A.-G. (E.P. 241,155, 16.4.25. Conv., 11.10.24).—Timber to be impregnated by the "kyanising" process is placed in tanks through which the mercuric chloride is circulated continuously from a supply tank at 40°, even distribution being ensured by transverse wooden grids in the steeping tanks. A comparatively short time is necessary for impregnation, and uniform and complete impregnation is obtained. B. W. Clarke.

Bituminous emulsions. Asphalt Cold Mix, Ltd., and F. Levy (E.P. 246,907, 3.11.24, 30.4 and 29.7.25).—Hot molten or liquid bituminous material (at a temperature of 90—110°) is agitated with up to 10% of a tanning agent, *e.g.*, tannic acid, as an emulsifier, and up to 100% of hot water, with or without the addition of an alkali. The addition of a dilute aqueous solution of an alkali increases the stability of the emulsion and enables a higher proportion of water to be used. The emulsion formed is stable and durable and can be readily poured and used in the cold state. B. W. Clarke.

Manufacture of cement. K. P. Billner. From M. Lantz (E.P. 247,097, 22.8.25).—Gypsum, or other suitable material containing calcium salts, is incorporated with cement clinker in two or three stages, 2—3% being added at each stage and ground thoroughly with the clinker. In this way a comparatively high percentage of gypsum can be added, reducing the setting time and increasing the compressive strength and yet reducing the volume expansion (Le Chatelier test). B. W. Clarke.

Manufacture of Portland cement from molten slag from gas producers and furnaces fired by pulverised coal. G. Polysius (G.P. 421,427, 20.4.22).—Lime, silica, ferric oxide, alumina, and gypsum are added, in the proportions required to form Portland cement, to the molten slag before it is tapped off from the slag chamber. The added substances may be introduced, together with fuel and air, through nozzles provided with several concentric outlets or through simple nozzles.
B. W. Clarke.

Binding together blocks for pavements, roads and the like. L. S. van Westrum (E.P. 247,238, 12.8.24 and 9.5.25).—A mortar for binding road or building materials together is composed of 20 pts. of cement, lime, plaster of Paris, or the like, mixed with 80 pts. of a bituminous soap which contains at least 50% of bitumen and which is made by saponifying fatty material in presence of bitumen (cf. E.P. 233,371; B., 1926, 14). The mortar may also contain 10—20 pts. of pulverised rubber (if rubber paving is being dealt with) or 10—20 pts. of sand or stone dust. A. W. Hothersall.

[Magnesium oxychloride cement] railroad tie. H. G. Jaeger (U.S.P. 1,568,507, 1.5.26. Appl., 4.10.22).—Railroad ties specially suitable for tropical climates are prepared by saturating a loose fibrous organic material with a phenolic preservative. The product is saturated with magnesium chloride solution and calcined magnesite added, and then a separately prepared finely powdered mixture of

silica and calcined magnesite is incorporated, the whole mixture being moistened to a plastic consistency with magnesium chloride solution.

S. Binning.

Manufacture of magnesia from crystalline magnesites for the production of Sorel cement. Austro-Amer. Magnesite Co. (Oesterr.-Amer. Magnesitges.) (Austr. P. 101,328, 22.5.23).—Definite proportions of finely ground and lump magnesite are mixed and heated for a prolonged period at a definite temperature above the decomposition temperature of magnesite. Tho product contains, in addition to highly basic magnesium carbonate which sets rapidly with solutions of magnesium chloride or magnesium sulphate, dead-burnt magnesia which sets very slowly and increases the stability of the cement.

B. W. Clarke.

Manufacture of porous building material for insulating purposes from coal slack etc. W. Neuhaus and E. Opderbeck (G.P. 412,071, 20.1.24, and 421,072, 16.2.24).—A mixture of coal washings, shale, etc. is ignited and burnt in specially constructed chambers without the addition of further fuel or the application of external heat. In order to maintain a sufficient temperature, oxygen or oxygen mixed with air is introduced under pressure. For the purpose of quick building the mixture is charged into the annular space between outer and inner refractory walls, and fired as described.

B. W. Clarke.

Manufacture of porous bricks or bricks containing metals or metal oxides. C. Ehrenberg, H. Wiederhold, C. Krug, M. G. Holsboer, K. Fischer, and Studienges. für Ausbau der Ind. (G.P. 422,715, 20.3.24).—The mass from which the bricks are formed is mixed with ammonium salts of humic acid, dried, and fired, a reducing atmosphere being used when it is desired to obtain bricks containing metals. B. W. Clarke.

Process for preparing cement. W. Kirchner, Assr. to Chem. Fabr. Grünau, Landshoff & Meyer A.-G. (U.S.P. 1,578,139, 23.3.26. Appl., 1.2.24).—See G.P. 405,844; B., 1925, 209.

Impregnation of wooden posts. Établ. P. Noé & Co. (E.P. 214,550, 12.10.25. Conv., 16.10.24).

Heating of heavy tars and bitumens in connexion with the treatment of roads. A. E. Ridgway (E.P. 249,304, 5.3.25).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Balanced reactions in steel manufacture. A. M'Cance (Trans. Faraday Soc., 1925, 21, 176—201).—The equilibrium constants of several reactions occurring in the manufacture of steel have been derived theoretically and the equations for iron and iron oxides have been evaluated. The Nernst heat theorem and Einstein's equation for the atomic heats of solids deduced from the quantum hypothesis have been applied in considering other elements, such as manganese and silicon, where experimental data and information on specific heats of many compounds involved are lacking. The percentage deoxidation of liquid steel at four different temperatures between 1500° and 1800° in the presence of varying amounts of manganese from 0·10 to 2·00% is given, and similar calculations have been made in the case of silicon. A steel originally saturated with FeO at 1500° is deoxidised to the extent of 81·7% if manganese is added in such amount that 0·5% remains in the mixture. The ratio of the deoxidising powers of manganese and silicon obtained theoretically agrees well with Brinell's figure of 5·2. Ordinary deoxidised steel contains 0·04—0·05% O, and it would be difficult to reduce this further by means of manganese or silicon. Only very small amounts of aluminium in excess of that required to effect deoxidation are required to maintain the deoxidised state, and for practical purposes the amount can be regarded as independent of temperature. Aluminium removes the whole of the free ferrous oxide, and the most economical method is to use a low-grade alloy containing about 5—10% Al. Silicon can be reduced from the slag by iron, but more is reduced by carbon. In open-hearth practice carbon is oxidised during the boil period through the intermediate action of ferrous oxide in the slag, and as soon as this falls silicon is reduced. On increasing ferrous oxide by the addition of ore the reduced silicon is oxidised and a cloud of minute inclusions is produced throughout tho bath. To minimise the amount of non-metallic inclusions the total quantity of ore should be added at one time, and the slag allowed gradually to exhaust itself. In passing from slags of high silica content to slags high in ferrous oxide the number of inclusions tends to diminish, this difference being less marked in cold than in hot charges.

M. Cook.

Effect of other elements on the migration of carbon in steel. E. G. Mahin, R. C. Spencer, and C. R. Hayner (Proc. Indiana Acad., 1925, 34, 177—180; Chem. Abstr., 1926, 20, 571).—An introductory paper. Cementation experiments are held to prove that solid carbon can dissolve in and migrate through iron. Cementation is more rapid from grey than from malleable iron into ingot iron, and the differences in the effect are ascribed to differences in carbon, manganese, or silicon content. Increasing the amount of silicide or of ferromanganese in a carburising mixture of grey iron drillings increased the tendency of iron to absorb carbon.

A. A. Eldridge.

Metallographic etching reagents. III. For alloy steels. E. C. Groesbeck (Sci. Papers U.S. Bur. Standards, 1925, 20, [518], 527—586).—Etching with acid reagents does not distinguish between any of the constituents found in alloy steels and ferro-alloys. By the ordinary immersion method with solutions containing an alkali two distinct etching effects are obtained, a chemical attack of the constituent by the alkali and oxidation of the constituent by nascent oxygen generated in the solution.

The oxidised compound is generally etched out, and the iron matrix remains bright in both cases, having been rendered passive by the alkali or the nascent oxygen. Iron carbide, iron tungstide, and the complex constituent of high-speed steels are attacked by the alkali, the other constituents remaining undarkened, while all are affected and darkened by nascent oxygen. The first three are distinguished from chromium carbide, tungsten carbide, or vanadium carbide by etching with a hot 10% solution of sodium or potassium hydroxide, alkaline sodium picrate, or alkaline potassium copper cyanide. Iron tungstide or the high-speed steel constituent are distinguished from carbides by etching in 10% sodium or potassium hydroxide or a mixture of 10% sodium hydroxide and hydrogen peroxide. In a hot sodium picrate solution iron tungstide is darkened in 1 min., whereas all the other constituents remain undarkened for at least 10 min. In electrolytic etching, using solutions of weakly dissociated acids and both alkali and metal salts of these acids, the iron matrix remains unattacked while the other constituents are etched. With an etching period of 3 min. the carbides of chromium, tungsten, and vanadium are attacked, while iron tungstide, iron carbide, and the high-speed steel constituent remain unaffected. Oxygen is formed at the anode, which is the specimen being etched, through a secondary reaction between water and the acid formed in the first stages of electrolysis and also by the discharge of hydroxyl ions at the anode. The various constituents, excepting iron carbide which is darkened by this treatment, cannot be distinguished from each other by heat tinting. By suitable etching methods and solutions it is possible to identify any of the constituents, but tungsten carbide and chromium carbide cannot be distinguished from each other, although the crystalline form of tungsten carbide, which usually occurs as triangular or rectangular shaped particles, aids in its identification.

M. Cook.

Heat-treatment of steel rails. L. Thibaudier and H. Viteaux (Rev. Mét., 1926, 23, 65—81).—At the Neuves-Maisons works steel rails are subjected to intermittent quenchings in cold water. The volume of water in the quenching bath is regulated according to the weight of metal, and means are provided above the bath for lowering or raising the rail (cf. E.P. 195,147; B., 1923, 505 A).

C. A. King.

Cementation of ferrous alloys with aluminium. J. Cournot (Compt. rend., 1926, 182, 696—698).—Different methods of cementation were tested, and the powdered ferro-aluminium process found the most satisfactory, ammonium chloride being used as a flux. Steel low in carbon content was used. Two layers were formed, viz., an outer one less than 0·1 mm. thick (composed of a solid solution containing $FeAl_3$), which is irregular, porous, and brittle, and an inner layer of composition ranging from 65 to 100% Fe, which forms a good protecting layer. The variation of efficiency of covering with temperature and time of cementation was tested by heating in an oxidising atmosphere. Micrographical examination was also carried out. A specimen cemented for 1 hr. at 900° had an inner protective layer 0·02 mm. thick, and resisted oxidation in an oxidising atmosphere at 1000° for 81 hr. After cementation for 8 hr. the inner layer was 0·37 mm. thick, and resisted oxidation for 240 hr. The constants in the equation of Weiss (Thèse, Paris, 1923, 85) representing the rate of formation of the inner layer of solid solution were calculated. For a layer 0·32 mm. thick, if θ = time in hours, and T = absolute temperature, $1/\theta = 6{\cdot}323 \times 10^{-4} + 1{\cdot}0059^{T}$.

W. Thomas.

Specific electrical resistance of tungsten wires. J. Tsukamoto (Nagoka Anniv. Vol., 1925, 409—412; Chem. Zentr., 1926, I., 1701).—The specific electrical resistance of drawn tungsten wire containing 1% Th is dependent upon the fineness of the tungsten powder from which the wire was made, on the time of sintering, and on the temperature of drawing. At 20° the specific resistance of a rod, 0·47 cm. square, is 6·1 microhms, which falls to a minimum of 5·7 microhms on drawing down to a diameter of 4·5 mm. Further drawing increases the resistance to 6·0 microhms at 0·35 mm. diameter, and to 7·1 microhms at 0·015 mm. diameter.

A. R. Powell.

Tensile strength of tungsten wires at high temperatures. R. Tajime (Nagoka Anniv. Vol., 1925, 413—416; Chem. Zentr., 1926, I., 1701).—The tensile strength of tungsten wires containing 1% Th and having a diameter of 0·076 mm. is as follows in kg. per sq. mm.: 310 at 300° abs., 200 at 800° abs., 70 at 1600° abs., 20 at 2000° abs., 9 at 2400° abs., 4·5 at 2800° abs., and 1·5 at 3150° abs. All the determinations were carried out *in vacuo*.

A. R. Powell.

Differential dilatometer with mechanical registration [for metals]. P. Chevenard (Rev. Mét., 1926, 23, 92—99).—The differential dilatometer designed for automatic registration by optical means (Rev. Mét., 1917, 14, 610) has been modified by replacing the concave mirror of the optical lever by a pointer placed perpendicularly to the plane of the three contact points. A pen attached to the end of this pointer marks the curve of differential expansion on a sphere, and mechanical means are used to translate this movement on to a single plane.

C. A. King.

Porosity and intensive corrosion. Experiments on commercial sheet zinc and other metals. U. R. Evans (J.S.C.I., 1926, 45, 37—44 T). The corrosion of commercial sheet zinc in chloride or sulphate solutions is of four types:—(1) Regional corrosion of the lowest portion (where oxygen soon becomes exhausted and which becomes anodic towards the part above); a sharp horizontal boundary separates this region from the unchanged area above and moves steadily upwards. (2) Point corrosion starting in pores opening on to the surface, which represent ideal "unaerated" (anodic) places. (3) Line corrosion along scratches. (4) Edge-point corrosion from points or edges. Corrosion along

pre-existing capillary channels, which are known to penetrate even ordinary sound metals, often causes marked internal rottenness. In rolled metal corrosion produces a great tendency to flaking owing to the undermining of the surface layers.

Effect of cyanogen compounds on the floatability of pure sulphide minerals. E. L. Tucker, J. F. Gates, and R. E. Head (Min. and Met., 1926, 7, 126—129).—The addition of lime to the pulp prevents almost completely the flotation of pyrites and reduces considerably the flotation of chalcocite, but in no way influences the flotation of bornite or chalcopyrite when a mixture of pine oil and potassium xanthate is used as the frothing agent. Addition of sodium cyanide in an amount not exceeding 0·5 lb. per ton of mineral further decreases the amount of pyrites floated but increases the amount of chalcocite floated when the alkalinity is relatively high. The "deadening" of pyrites by lime and cyanide is more efficient and lasting than that caused by lime alone and is caused by the adherence of agglomerated particles to the surface of the pyrites. Microchemical examination of treated pyrite particles showed the presence of ferric iron on the surface.
A. R. Powell.

Rate of solution of base metal amalgams in acids. W. Fraenkel (Korrosion u. Metallschutz, 1925, 1, 203—206; Chem. Zentr., 1926, I., 1703—1704).—The rate of solution in acids of the alkaline-earths metals, aluminium, magnesium, and beryllium from amalgams is expressed by the equation $dv_t/dt=K(v-v_t)$, where v is volume of hydrogen disengaged by complete solution of the metal and v_t is the volume disengaged in time t. There appears to be no relation between the coefficient of diffusion and the constant K or the potential for any of the metals tested. The concentration of the above metals in the amalgam has a distinct influence on the rate of dissolution of the metal in acid, but with alkali metals this is not the case. The difference is probably due to the presence of dissolved and slightly dissociated intermetallic compounds in the latter case.
A. R. Powell.

Crystal growth in recrystallised cold-worked metals. W. Feitknecht (Inst. Metals, Mar., 1926. Advance copy, 35 pp.).—Crystal growth in recrystallised cold-worked aluminium sheets is faster the more severe the deformation prior to recrystallisation. Crystals attain only a moderate size in severely worked material but the size increases with decreasing deformation, 30% reduction in thickness yielding very large crystals. Growth only commences after heating for a certain period, during which time little change takes place. This period is longer the less severely the material has been worked. Large crystals commence to grow from the surface. No marked change takes place below a certain limit of deformation and the limit below which no large crystals grow is about 30% at 550° and 10% at 630°. The minimum grain size is practically the same in sheets of different thickness and mechanical treatment which have been heated to the same temperature and in which growth is completed. A short heat treatment at a higher temperature causes growth to be much slower on annealing at a lower temperature, and if the temperature of the first annealing is sufficiently high no considerable growth occurs at the lower temperature. A short previous annealing at a higher temperature or a longer one at a lower temperature raises the limit of deformation below which no large crystals can grow. Grains which have grown can subsequently decrease in size and remain stable at a smaller size or disappear entirely. Growth is not confined to large grains and the process is very discontinuous. The grain size after complete recrystallisation is much larger in very pure aluminium than in a less pure variety and growth is much faster. As in pure aluminium, crystal growth in very pure silver is faster the more severely the sheets have been worked, but the final grain size is much finer than in aluminium heated at a corresponding temperature. Difference of internal energy of crystal grains is considered to be the main cause of crystal growth. Insoluble impurities greatly raise the resistance to growth. The two main conditions necessary to cause growth of very large crystals are a not too great amount of remaining strain and a certain amount of resistance which is slowly removed on heating for long period.
M. Cook.

Softening of strain-hardened metals and its relation to creep. R. W. Bailey (Inst. Metals, Mar., 1926. Advance copy, 14 pp.)—An examination of data of various authors on the softening of several strain-hardened non-ferrous metals and alloys supports the view that the relation between the time T to produce a specific softening and the temperature θ at which it takes place is of the form: $T=T_0e^{-b\theta}$, where T_0 is the time required to soften at zero temperature, b a constant varying with different metals. This law gives a straight line upon the log (time)te-mperature diagram and the curvature of the line plotted from experimental data is due to the influence of the heating up period and is much more marked at high than at low temperatures. The three stages of creep in the failure of a test-piece in tension under prolonged application of stress at a constant temperature are explained on the balance of the rate of production of strain-hardening by distortion and the rate of its removal by thermal action. If the test is made at two different temperatures, and assuming the physical properties of the metal to be the same at these temperatures, the times to failure should be proportional to the times for softening of strain-hardened material, and thus the duration would be connected with the temperature by the same law as that for the softening of a strain-hardened metal, *i.e.*, the value of L, the length of life at temperature θ, would be $L=L_0e^{-b\theta}$.
M. Cook.

Malleability and metallography of nickel. P. D. Merica and R. G. Waltenberg (Tech. Papers U.S. Bur. Standards, 1925, 19, [281], 155—182).—See B., 1925, 175.

Physical chemistry in steel making. R. HADFIELD (Trans. Faraday Soc., 1925, **21**, 172—175).—See B., 1925, 550.

Reactions of basic open-hearth [steel] furnace. T. P. COLCLOUGH (Trans. Faraday Soc., 1925, **21**, 202—223).—See B., 1925, 550.

Chemical reactions of the basic electric [steel] process. F. T. SISCO (Trans. Faraday Soc., 1925, **21**, 224—239).—See B., 1925, 550.

Equilibria in systems involving ferrous oxide. J. B. FERGUSON (Trans. Faraday Soc., 1925, **21**, 240—242).—See B., 1925, 551.

Slag reactions. P. M. MACNAIR (Trans. Faraday Soc., 1925, **21**, 243—248).—See B., 1925, 551.

Function of ferric oxide in acid and basic open-hearth slags. J. H. WHITELEY (Trans. Faraday Soc., 1925, **21**, 249—254).—See B., 1925, 551.

Physico-chemical phenomena from melt to ingot. A. L. FEILD (Trans. Faraday Soc., 1925, **21**, 255—267).—See B., 1925, 550.

See also A., April, 340, **Effect of rolling on crystal structure of aluminium** (OWEN and PRESTON). 344, **Gold–nickel alloys** (FRAENKEL and STERN); **Metal-pairs forming a continuous series of mixed crystals** (VAN LIEMPT); **Solubility of hydrogen in tin and aluminium at high temperatures** (BIRCUMSHAW). 349, **Permeability of nickel to hydrogen** (LOMBARD). 356, **Inter-metallic compounds. Calcium–tin and magnesium-tin alloys** (HUME-ROTHERY); **Diagram of state of calcium and mercury** (EILERT). 357, **System iron–chromium–carbon** (MEIERLING and DENECKE). 358 **Phase equilibria of sulphates** (Jänecke); **Reduction equilibria for system zinc oxide–carbon monoxide** (MAIER and RALSTON).

Combustibility of blast-furance coke. SHERMAN and KINNEY.—See II.

Overcoming plating troubles. BERNARD.—See XI.

Colloidal metals. HUGOUNENQ and LOISELEUR.—See XX.

PATENTS.

Manufacture of alloys [containing silicon, titanium, or zirconium]. R. W. STIMSON and W. BORCHERS (E.P. 247,634, 6.11.24).—Alloys of silicon, titanium, and zirconium with any number of miscible metals, *e.g.*, iron, chromium, manganese, are produced by smelting in an electric furnace a mixture of carbonaceous reducing agent, slags or natural products rich in alumina, sand or slags rich in silica, zirconia, and/or titania, calcareous materials, such as fluorspar or limestone, and metalliferous ores or by-products containing metals of the iron group, manganese, chromium, copper, or the like, the mixture being so apportioned that the resulting slag contains silica, alumina, and lime and magnesia in such proportions that the chemical equivalent of the silica exceeds that of the other acid components, the sum of the chemical equivalents of the alumina, lime, and magnesia exceeds 75% of that of the silica, and the chemical equivalent of the alumina exceeds 85% of the sum of those of the lime and magnesia. For producing a chromium-silicon-iron alloy an aluminous slag is melted on the carbonaceous hearth of an arc furnace and briquettes of low-grade chromium slag and chromite bonded with petroleum tar are fed in alternately with briquettes of silicious material, lime, and tar; the temperature is maintained at 1700—1750° until a grey slag of the above nature is produced containing less than 1% Cr_2O_3 and 1% FeO. The resulting alloy contains for example, 24% Si, 43% Cr, 0·4% C, and the remainder iron, and is suitable for the direct production of low-carbon chromium steel (non-rusting steel); to this end it is added together with chromite to a steel bath fused under a calcareous flux in an arc furnace with a non-reducing hearth, whereby further chromium is reduced from the ore and the silicon enters the slag as silica, together with much chromic oxide. The steel so produced is practically free from sulphur and phosphorus, while the slag comprises a low-grade source of chromium and silicon for the production of further quantities of silicon-chromium alloy for use again in the process. The poor slag produced in the first step is useful for the production of cement. Low-grade manganese ores containing much phosphorus and iron are smelted with a silicious flux to produce an iron alloy rich in phosphorus and a highly manganiferous slag which is smelted as above for the production of a silicon-manganese-iron alloy for use in steel making and a slag for cement manufacture. The iron-phosphorus alloy is refined by the basic open-hearth process to obtain steel and a slag rich in phosphorus for fertilisers. Cuprosilicon alloys for use as deoxidisers may be made in a similar manner to the chromium-iron-silicon alloys except that copper oxide replaces the chromium material in the charge. A. R. POWELL.

Manufacture of [steel] alloys. R. W. STIMSON (E.P. [A] 247,635, 6.11.24, and [B] 247,876, 6.11.24, and 23.2.25).—(A) Alloy steels of the non-rusting type containing more than 50% of iron with nickel and/or cobalt and more than 3% of an additional alloying element are made in the following way. Clean open-hearth steel scrap is fused in a basic-lined arc furnace with lime and sufficient manganese ore to give a strongly basic slag and oxidise the sulphur and phosphorus, but not all the carbon in the metal; from time to time further lime with borax and/or fluorspar are added to render the slag very fluid. The slag is removed, the metal covered with lime and fluorspar, and a mixture of iron–chromium–silicon alloy (containing, for example, 22% Si and 51% Cr, cf. E.P. 247,634, preceding), magnetic iron ore or roll scale sufficient to oxidise the silicon, and lime sufficient to combine with the silica formed is added, suitable fluxes being charged in as required. The

slag is again removed when the reaction is finished and replaced by a desulphurising slag containing less than 2% C as calcium carbide, after which a small amount of ferromanganese is added to ensure thorough deoxidation of the metal and efficient removal of gases. If an addition of copper is required it is made prior to the calcium carbide slag wash, whereas nickel, cobalt, molybdenum, etc. should be added to the original bath of fused metal, or scrap containing these elements may be used instead of plain carbon steel. (B) The alloying of the desired element (*e.g.*, chromium) is effected by adding to the molten steel bath, after removal of the phosphorus, a mixture of an ore of the desired element, a non-carbonaceous reducing agent, and a flux, *e.g.*, a mixture of chromite, ferrosilicon containing 90% Si, and burnt lime, together with suitable additions of borax, fluorspar, iron, manganese and titanium oxides to maintain a fluid slag. After removal of the slag the bath is purified as described under (A). A. R. POWELL.

Preventing adherence of cast metal [steel] to the mould. F. VON BICHOWSKY (U.S.P. 1,570,802, 26.1.26. Appl., 22.4.24).—Finely divided titanium, silicon, aluminium, boron, or zirconium nitride, preferably in the crude state mixed with iron, as obtained by the method described in U.S.P. 1,391,147–8 1,408,661, and 1,415,280 (B., 1921, 770 A; 1922, 294 A, 463 A) is made into a paste with water, with or without addition of a binding agent, such as molasses or sodium silicate, and used to coat moulds for steel or the like. The dried lining is permeable to gases from the molten metal, and the casting readily comes away from the mould and requires only simple sand-blasting before machining. T. S. WHEELER.

Manufacturing open-hearth steel. J. D. JONES and E. E. LITZ (U.S.P. 1,573,004, 16.2.26. Appl., 6.12.21).—A bath of metal in an open-hearth furnace is heated by high-velocity flames from a number of burners spaced equal distances apart around a circle and radiating from the furnace wall towards the centre of the furnace. C. A. KING.

Manufacture of cast-iron. J. C. HENDERSON, Assr. to DRIVER-HARRIS CO. (U.S.P. 1,573,937, 23.2.26. Appl., 22.4.25).—Cast-iron containing a definite amount of chromium and nickel is made by adding to a molten mass of iron in a ladle the requisite amount of a solid alloy containing nickel with a smaller amount of chromium and iron and sufficient carbon to give a low melting point. A. R. POWELL.

Process for treating iron. W. J. DIEDERICHS and A. HAYES (U.S.P. 1,574,374–7, 23.2.26. Appl., [A] 21.8.22, [B—D] 6.8.25).—Castings made from white iron are subjected to the following heat treatment:—(A, C). The casting is heated above the critical temperature for a period between 15 min. and 5 hrs. or more, but only until the iron carbide is practically all absorbed into solid solution, which in practice means that combined carbon is reduced to 0·9%; the casting is then cooled to a temperature near the critical point, and finally, in (A), cooled to room temperature, or in (C), cooled at a rate not less than 7° per hr. (B, D). The casting is heated above the critical temperature, then cooled considerably below that temperature, reheated sufficiently to allow structural and chemical changes to take place, and finally in (B), cooled at an extremely slow rate, about 5° per hr., or in (D), cooled sufficiently to prevent further chemical and structural changes. B. M. VENABLES.

Reduction of iron ore. P. FARUP, Assr. to NORSK HYDRO-ELEKTRISK KVAELSTOF-A./S. (U.S.P. 1,574,382, 23.2.26. Appl., 10.5.22).—Iron ores are reduced in an atmosphere of carbon monoxide, and the product is cooled in an atmosphere containing only a relatively small proportion of this gas. A. R. POWELL.

Non-magnetic steel. E. BECKER, Assr. to POLDIHÜTTE (U.S.P. 1,574,782, 2.3.26. Appl., 25.5.25).—An alloy steel containing 6—15% Mn, 17—4% Ni, and an appreciable quantity, not exceeding 10%, of a metal having similar properties to chromium, has a high yield point and elongation in its normal condition. A. R. POWELL.

Furnace for the production of iron sponge. HÖGANÄS-BILLESHOLMS AKTIEBOLAG (F.P. 599,185, 8.6.25).—The material is charged into a number of honeycombed retorts which are placed in a reverberatory furnace so that the heating gases play around and between them. A. R. POWELL.

Removal of oxides from ferrous metal. R. PORTER and J. C. WHETZEL, Assrs. to AMER. SHEET AND TIN PLATE CO. (U.S.P. 1,572,848, 9.2.26. Appl., 27.8.24).—Articles of ferrous metal are immersed in an alkaline solution to break down the resistance of the oxide to acids, and then in an acid pickling bath to remove the oxide. L. A. COLES.

Welding processes [using atomic hydrogen] and apparatus therefor. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of R. PALMER (E.P. 237,901, 23.7.25. Conv., 30.7.24; cf. E.P. 237,898; B., 1926, 198).—A stream of hydrogen is passed through an electric arc and thereby dissociated into atomic hydrogen, and the heat of re-formation of molecular hydrogen is utilised to weld metals. The welding torch comprises inclined electrodes with a nozzle for a localised stream of hydrogen within the angle between the electrodes. In addition to the localised stream of dissociated hydrogen, additional hydrogen may be supplied through a "welding head" or cover, to exclude air entirely from the weld. Electrodes of refractory metals such as tungsten may be used, and the reducing action of the atomic hydrogen is so great that easily oxidised metals or alloys, such as ferro-chrome, may be used in the weld. B. M. VENABLES.

[Bearing metal] alloys containing lead and tungsten. H. FALKENBERG (E.P. 247,687, 8.12.24).—Tungsten is melted with an equal weight of lead-antimony alloy and a small amount of iron, the

mixture is further diluted with lead-antimony alloy, and then melted with a tin-antimony-copper-lead alloy or a lead-iron-zinc alloy. For example, a mixture of 50% of tungsten, 49·5% of antimonial lead, and 0·5% of iron is melted, diluted with three times its weight of antimonial lead, and cast. One part of this alloy is then melted with 100 times its weight of an alloy containing 67·2% Pb, 18% Sb, 13% Sn, and 1·8% Cu, or of an alloy containing 91·5—96% Zn, 0·5—5% Fe, and 3·5% Pb with, if desired, small amounts of copper and aluminium. The antimonial lead used contains 18—20% Sb.

A. R. Powell.

Aluminium-copper alloy. Z. Jeffries and R. S. Archer, Assrs. to Aluminum Co. of America (U.S.P. 1,572,487, 9.2.26. Appl., 29.4.24).—Castings of an aluminium alloy containing between 3 and 5·5% Cu are heated to 500—540° for at least 7 hr. and then cooled. M. Cook.

Aluminium-silicon alloy. Z. Jeffries and R. S. Archer, Assrs. to Aluminum Co. of America (U.S.P. 1,572,488, 9.2.26. Appl., 29.4.24).—Castings of an aluminium alloy containing substantial amounts of silicon and zinc are heated at a temperature slightly below the melting point of the eutectic until the silicon particles have altered sufficiently to improve the physical properties of the alloy.

M. Cook.

Aluminium alloy. A. Pacz, Assr. to Aluminum Co. of America (U.S.P. 1,572,502, 9.2.26. Appl., 12.9.19).—An alloy containing aluminium, copper, and silicon in the proportion of 70, 15—27½, and 2½—15 pts. by weight respectively is claimed.

M. Cook.

Composition of matter. [Die-casting alloy.] A. Pacz, Assr. to Aluminum Co. of America (U.S.P. 1,572,503, 9.2.26. Appl., 31.5.21).—A die-casting aluminium alloy contains nickel and between 2·5 and 10% Si. M. Cook.

Nickel alloy. P. D. Merica, Assr. to Internat. Nickel Co. (U.S.P. 1,572,744, 9.2.26. Appl., 26.6.23).—In the manufacture of nickel alloys the principal melt is deoxidised and then mixed with molten aluminium. M. Cook.

Dental alloy. C. C. Vogt, Assr. to L. S. Smith and Sons Manuf. Co. (U.S.P. 1,574,714, 23.2.26. Appl., 26.12.22).—A dental alloy comprises silver and tin in amounts which will readily form a suitable hardening amalgam with mercury, together with a small amount of tungsten. A. R. Powell.

Removing silica from [acid] ore leaches. M. Hosenfeld, Assr. to Siemens & Halske A.-G (U.S.P. 1,571,054, 26.1.26. Appl., 16.1.25).—Acid leaches from zinc ores or the like containing precipitated silica, which tends to gel and prevent filtration, are treated with calcium or barium sulphate or other pulverulent insoluble substance in the proportion of 5 g. per litre, and heated to 70°. The mixture can then be readily filtered. A portion of the insoluble residue can be used in the treatment of another batch of leach liquor, while the remainder may be mixed with phosphates and used as a fertiliser; the finely divided silica interacts with the phosphates converting them into soluble compounds.

T. S. Wheeler.

Continuous ore-treating furnace. W. P. Jobson, Assr. to H. S. Souder (U.S.P. 1,572,291, 9.2.26. Appl., 15.8.24).—Ore is fed and removed from a retort intermittently and fumes which are drawn off are washed to precipitate suspended solid matter. M. Cook.

Smelting ores of volatile metals. Smelting volatilisable metals. W. A. Ogg (U.S.P. 1,573,013–5, 16.2.26. Appl., [A] 7.2.25, [B] 18.5.25, [C] 25.5.25). —(A) A mixture of crude zinc-bearing material and reducing fuel is distilled in a retort in excess of the capacity of the condenser. Vapours discharged from the condenser, representing a considerable fraction of the total metal values, are recovered by deposition, while spelter is recovered in the condenser to its full capacity. (B) In the chloridising reduction of zinc ores under similar conditions to those specified in (A), fumes leaving the condenser are deposited, leached with water, and the solution mixed with a fresh charge of crude material and fuel to make briquettes for a subsequent volatilising operation. (C) The process is similar to that described under (B), but the solution obtained by leaching the fume product is treated to precipitate the zinc, the residual chloride solution being used with a further charge of ore. C. A. King.

Separation and recovery of copper, tin, and lead content of brass or bronze secondary metals [scrap] and residues. T. Lewin (U.S.P. 1,574,043, 23.2.26. Appl., 24.8.25).—The material is smelted to remove iron and non-metallic impurities, zinc oxide being recovered in a bag plant. The metal is cast into anodes and electrolysed for the production of cathode copper and lead-tin anode slimes, the latter being subsequently smelted for the recovery of the constituent metals.

A. R. Powell.

Collecting, refining, and utilising by-products from metals treated in retorts. H. W. Tuttle (U.S.P. 1,574,137, 23.2.26. Appl., 5.9.22).—The fumes are passed through a filter and passageway closed to the admission of air, and means are provided for removing the solids precipitated on the filter and for collecting and utilising the gases without oxidation taking place. A. R. Powell.

Protective coating for magnesium. L. J. Keeler, Assr. to Amer. Magnesium Corp. (U.S.P. 1,574,289—90, 23.2.26. Appl., 22.1.23).—Magnesium and its alloys are coated (A) by bringing the metal into contact with nascent fluorine, or (B) by using the metal as an anode in a solution of alkali chromate.

A. R. Powell.

Depositing silver. B. Bart (U.S.P. 1,574,544, 23.2.26. Appl., 23.9.21).—A solution of a silver

salt is mixed with a reducing agent and set aside in absence of air until part of the silver is precipitated in a metallic form. The mixture of solution and finely-divided metal is then sprayed on the surface to be silvered. A. R. POWELL.

Separation of metals [copper and nickel]. S. GIERTSEN, Assr. to KRISTIANSANDS NIKKEL-RAFFINERINGSVERK (U.S.P. 1,575,160, 2.3.26. Appl., 26.9.21).—Copper is precipitated from solutions of copper and nickel salts by treatment with bessemerised copper-nickel matte containing less than 10% S. A. R. POWELL.

Recovery of zinc from ores. P. J. LEEMANS (F.P. 598,090, 23.8.24).—Low-grade zinc ores are heated with a reducing agent such as coal, and, if they contain sulphur, also with copper, to such a temperature that the zinc and part of the lead are volatilised and collected as oxides. The product is separated by treatment with ammonium carbonate solution, which dissolves the zinc. A. R. POWELL.

Preparation of gold [from mercury]. SIEMENS & HALSKE A.-G. (F.P. 598,140, 7.5.25. Conv., 8.5.24).—A high-tension discharge is passed between two mercury electrodes one of which forms an annular ring around the other. In the presence of catalysts such as silver, part of the mercury (probably the isotope of at. wt. 197) is said to be converted into gold (cf. E.P. 233,715, B., 1925, 996). A. R. POWELL.

Production of steel. A. BRÜNINGHAUS (E.P. 249,186, 10.12.24).—See U.S.P. 1,551,465; B., 1925, 885.

Reduction of oxide ores. Y. A. DYER (E.P. 248,633, 10.8.25).—See U.S.P. 1,556,316; B., 1925, 963.

Wet magnetic separation. G. ULLRICH, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,576,690, 16.3.26. Appl., 28.9.23).—See E.P. 220,196; B., 1924, 875.

Manufacture of thin sheets of tungsten, tantalum, molybdenum, and other refractory metals. GEN. ELECTRIC CO., LTD., Assees. of PATENT TREUHAND GES. FÜR ELEKTRISCHE GLÜHLAMPEN M.B.H. (E.P. 247,507, 27.5.25. Conv., 11.2.25).

Penetrators for testing penetration hardness [of metals]. C. H. WILSON (E.P. 249,446, 7.12.25).

Separating arsenic and/or antimony and tin. (U.S.P. 1,575,217).—See VII.

Recovering sulphur dioxide from smelting furnace gases. (Can. P. 248,607).—See VII.

XI.—ELECTROTECHNICS.

Overcoming plating troubles. H. C. BERNARD (Abrasive Ind., 1925, 6, 380—381; Chem. Abstr., 1926, **20**, 552).—An alkaline bath produces dark and clouded deposits of nickel; when the bath is too acid, the deposits are hard and brittle. Pitted deposits may arise from the use of a bath in which sedimentation of any solid material has not first taken place, or from the presence of insufficient metal in solution. Too high current densities give a dark, rough deposit, whereas that obtained with too low current densities is easily cut through when buffed. Copper yields a blistered or peeled deposit if too much cyanide is present, whereas with insufficient cyanide the anodes become coated and the deposit has a dark, muddy appearance. The various appearances of a brass deposit are considered in relation to the conditions of deposition. Operating conditions are given for the above and for silver baths. A. A. ELDRIDGE.

Effect of atmospheric humidity on the dielectric losses and power factors in fibrous insulating materials. S. SETOH and Y. TORIYAMA (Sci. Papers Inst. Phys. Chem. Res., 1926, **3**, 283—323).—In order to measure the dielectric losses in insulating materials, a quadrant electrometer free from electrostatic controlling force was used as an electrostatic wattmeter and details for its adjustment and calibration are given. Samples of insulating materials comprising different kinds of paper, calico, cloth, silk, some impregnated, others not, were tested. The humidity of the atmosphere was regulated by suspending the samples in a desiccator containing pure sulphuric acid of known concentration, the whole apparatus being placed in a thermostat kept at 30°. The dielectric losses increase at first slowly and then more rapidly with increasing humidity. Impregnated materials show generally smaller variations of losses with humidity, probably because the pores are more or less closed. The power factor, *i.e.*, the power losses in the samples, reaches a value of unity at about 70—80% humidity for untreated and also for imperfectly treated materials. Proper impregnation reduces the losses at high humidity. Hysteresis phenomena are observed when the humidity is increased from a low value and then decreased again, the dielectric losses when the humidity is decreasing being greater than when it is increasing. The dielectric losses under high humidity increase with the frequency first slowly, then a little more rapidly, and then again slowly as the frequency is increased. When the humidity is low, the change of losses with frequency follows a straight-line law. The power factor under high humidity decreases as the frequency is increased, while it remains almost constant under low humidity. The change of losses with time is much smaller for alternating than for direct current, and the final value is attained more rapidly. The losses vary with a definite power (varying between 1·9 and 2·45 for different humidities) of the voltage applied to the material for a constant humidity. The various ways in which moisture may affect the dielectric characteristics of the insulating materials are discussed, and an equivalent circuit, consisting of a high resistance connected in parallel with series-connected resistance and capacity, is shown to represent the actual variations of losses and power factor with frequency. M. E. NOTTAGE.

Electrical resistance of tungsten wires. Tsukamoto.—See X.

Patents.

Carbons or electrodes for the formation of ultra-violet rays for therapeutical purposes. M. A. and V. Arnone (E.P. 247,831, 11.7.25).—Carbons for producing arcs emitting ultra-violet rays for therapeutic purposes contain the following constituents in the proportions stated:—graphite 80 pts., retort carbon 5, lampblack 1, vegetable carbon 4, calcium (Ca) 5, magnesium (Mg) 3, strontium (Sr) 0·9, iron (Fe) 0·9, rare earths (thorium and uranium) 0·09, fluorine (F) 2·50, silica (SiO_2) 0·1 pt., aluminium (Al), sodium (Na), and potassium (K) traces. Constituents giving these elements are pulverised, mixed well by shaking, enough benzol added to form a thick paste, and the paste worked for some hours until it is homogeneous, after which it is strained and compressed into sticks at 200 atm. The sticks are dried by exposure to the air and afterwards baked, preferably in a Hoffman's oven. The baked carbons are cleaned, soaked in gum arabic as a protection against humidity, and dried by exposure to air. M. E. Nottage.

Thermoelectric element. O. Hermann, Assr. to Thermo Electric Battery Co. (U.S.P. 1,572,117, 9.2.26. Appl., 4.5.25).—An alloy of Ni 81%, Mo 17%, and V 2%, for use as the negative element of a thermo-couple, possesses the advantages of high e.m.f., low temperature coefficient of resistance, and a relatively small thermal conductivity. E. S. Kreis.

[Removing air bubbles from electrodes of] electrolytic apparatus. R. D. Mershon (U.S.P. 1,572,403, 9.2.26. Appl., 18.9.19).—An electrolytic cell is connected with an external pump which withdraws electrolyte and air from the cell and discharges these again into the cell beneath the electrodes, thus causing rapid movement of the electrolyte. If the electrodes are placed close together, or, where this is not feasible, are separated by perforated non-conducting plates, the agitation is increased. This may also be brought about by heating the electrolyte or allowing it to become heated by virtue of its own internal resistance. In such a case a layer of non-volatile paraffin is spread over the surface so that ebullition takes place regularly within the body of the electrolyte. The evolved vapours are condensed and returned to the cell. E. S. Kreis.

Composition for storage-battery electrodes. H. G. Weir, Assr. to Prest-O-Lite Co. (U.S.P. 1,572,586, 9.2.26. Appl., 6.11.23).—A paste is prepared with lead-containing sulphatable material and a lead compound of lower density than the active material to be prepared, the proportion of this compound being varied in accordance with the physical and chemical characteristics of the sulphatable material. The composition is placed on a support and converted into active material. H. Moore.

Electrical purification of gases. H. Edler (U.S.P. 1,574,237, 23.2.26. Appl., 9.2.25).—Bars of angular shape in cross-section serve as collecting electrodes and flat bands arranged with their narrow edges facing these are used as sparking electrodes. D. G. Hewer.

Electrical precipitation [of suspended particles from gases]. E. Hopkinson (U.S.P. 1,575,165, 2.3.26. Appl., 4.6.24).—The gas is passed through a long vertical flue containing an active electrode suspended in the middle throughout its length and two movable collecting electrodes one on each side of the central electrode. These are arranged on an endless band and move upwards from the bottom nearly to the top of the flue. A. R. Powell.

See also pages 346, **Preventing deposition of scale** (F.P. 599,188). 360, **Alkali phosphates** (U.S.P. 1,572,846). 368, **Welding processes** (E.P. 237,901). 369, **Recoving copper, tin, and lead** (U.S.P. 1,574,043); **Coating magnesium** (U.S.P. 1,574,290). 370, **Gold from mercury** (F.P. 598,140).

XII.—FATS; OILS; WAXES.

Chemical nature of fats. III. Significance of the difference between the upper iodine value and iodine value of a fat; differential iodine value. B. M. Margosches and H. Fuchs (Ber., 1926, 59, 375—376; cf. B., 1925, 410, 600).—The iodine value of an oil or fat varies with different specimens of the same material between certain limits. The upper iodine value varies in an exactly parallel manner, so that the difference between the two values (differential iodine value) also varies only within narrow limits and is characteristic of the oil or fat. The oils examined included olive, arachis, rape seed, almond, sesamé, cottonseed, linseed, tung, soya bean, sunflower, poppy, castor, whale, liver, fish, sardine, porpoise, coconut, and palm kernel oils. H. Wren.

Tung oil. E. Fonrobert and F. Pallauf (Chem. Umschau, 1926, **33**, 41—51).—The numerous researches on tung oil have failed to give satisfactory reasons for the gelatinisation which occurs on heating and for the characteristic cloudy "matted" or "webbed" surface, resembling crocodile skin, formed on dried films of this oil. Reference is made to the work of Eibner, Merz and Munzert (B., 1925, 477), Marcusson (B., 1923, 938A), and Wolff (B., 1924, amongst others, but the authors do not consider that the characteristic properties of this oil are due solely to colloidal phenomena. Tung oil consists largely of the triglyceride of elæostearic acid and it is in the very reactive double bonds of this acid that the authors seek the clue to the properties of the oil. Graphic formulæ are set out showing the possibilities of intramolecular and extramolecular polymerisation of the glycerides of this acid, and a conclusion, supported by much experimental data, is reached that the reactivity of the partial valencies of elæostearic acid leads to the formation of molecular complexes. According to their graphical arrangement four isomerides for elæostearic acid are possible, a bi-trans, a bi-cis, trans-cis and a cis-trans form; one of these may be the β-form of the acid, one is probably an unknown

liquid acid, and the other two, which differ but slightly, are probably the α-form of the acid. The recently published work of Böeseken and Ravenswaay (B., 1925, 813) indicating that elæostearic acid is an isomeric linolenic acid with 3 conjugated double bonds is briefly referred to by the authors who point out that such a view of the constitution of elæostearic acid allows of more than 4 isomers. H. M. LANGTON.

Oil-containing seeds of *Datura alba*, Nees. H. DIETERLE (Arch. Pharm., 1926, 264, 140—164).—The seeds of *Datura alba*, Nees (found in Florida), an illustrated anatomical description of which is given, yield about 13% of oil on extraction. The oil has d^{15} 0·9250; n_D^{40} 1·5144; acid value, 20·35; saponif. value, 192·8; Reichert-Meissl value, 2·41; Polenske value, 0·41; iodine value, 98·36; Hehner value, 93·6; acetyl value, 10·2. The oil has been exhaustively examined using, among others, the methods of Farnsteiner (B., 1898, 804) and Heiduschka and Lüft (B., 1919, 426A), with the following results: 2·9% of the oil is unsaponifiable; the remainder contains glycerides of the following acids, the proportions of which are given as percentages of the original oil. Palmitic and other saturated acids, 6·18%; caproic acid 1·00%; α-linoleic acid, 23·55%; β-linoleic acid, 2·92%; oleic acid, 60·86%. A fraction was obtained (m.p. 55—56°, magnesium salt, m.p. 136—140°) which corresponded in all respects with daturic acid (Gérard, B., 1890, 1137); it was found impossible to decide whether or not this was a mixture (cf. Heiduschka and Lüft, *loc. cit.*). W. A. SILVESTER.

Oxidation and polymerisation of vegetable oils and their fatty acids. G. PETROV and A. DANILOVITSCH (Z. Deuts. Oel- & Fett-Ind., 1925, 45, 669—671, 688—689, 733—705, 723—724).—In the course of an investigation on the preparation of oils of high viscosity the influence of blowing and heating on sunflower oil was particularly studied. By the distillation of the oil at 270—310°, about 20% of clear yellow fatty acids pass over, the highly viscous residue being likewise yellow, but free from any resinous substances. The conclusion is reached that sunflower oil is particularly suitable for the preparation of highly viscous lubricants. Polymerisation is effected with greatest rapidity at about 300°, in practice by the use of superheated steam under diminished pressure. The oil thereby also suffers partial hydrolysis and fatty acids are removed. The thickened oil is practically free from fatty acids and has an Engler viscosity of 6·44 at 100°.
H. M. LANGTON.

Determination of fatty acids in fats for customs purposes. H. HELLER (Z. Deuts. Oel- & Fett-Ind., 1926, 46, 148).—The Czecho-Slovakian customs imposes a duty on oils and fats containing more than 50% of free fatty acids as determined by the following test. Five g. of fat are melted and shaken with 50 c.c. of alcohol, and a few drops of phenolphthalein are added to the alcoholic extract, followed by 5 c.c. of potassium hydroxide solution (65·45 g. per litre). Samples which give a pink colour persisting for at least 15 sec. are admitted duty-free. It is pointed out that, taking the mol. wt. of oleic acid as a mean of that for the various fatty acids in commercial oils and fats, this method passes samples that contain less than 33% of free acid only, instead of the 50% allowed. A. R. POWELL.

Perfumed Marseilles soaps. G. DE BELSUNCE (Bull. Mat. Grasses, 1925, 288—293.)—The characteristic odour of coconut oil persists clearly even after saponification, and the author has studied the effects on Marseilles toilet soaps, made from coconut oil as a base, of various essential oils and perfumes added to the soap with the object of masking this odour. Samples of such soap were perfumed and the cakes examined after an interval of one week and again after an interval of three years. At the end of this latter period the original odour of coconut fat had not returned and the samples had not suffered rancidity, notwithstanding exposure to air and to temperature changes. Tables are given showing the amounts of the various natural perfumes necessary to mask completely for three years the coconut odour and the changes undergone in that time by the perfumes themselves are tabulated. Apparently the perfumes have a specific reaction different for different species and generally they act somewhat as resin in hindering rancidity. H. M. LANGTON.

Lubricating oils. SWOBODA.—See II.

Lubrication problem. VON DALLWITZ-WEGNER.—See II.

***X*-Ray spectrographic investigations on lubricants.** TRILLAT.—See II.

PATENTS.

Extracting fat from bones. J. HJORT (E.P. 247,058, 4.6.25).—The fat from the bones of whales and other large marine annimals is extracted by breaking the outer layer of the bones, whereby the inner spongy mass of bone is reduced to a pulpy condition and the fat can be drained off or separated by pressing or other meçhanical means. The process can be operated at ordinary temperature without previous boiling of the bones. The best results are obtained at low temperature, when the formation of oil emulsion in glue solution is avoided.
H. M. LANGTON.

Catalysts for hydrogenation (G.P. 424,067).—See I.

Extracting fat from bones (E.P. 247,347).—See XV.

Glycerophosphates (G.P. 421,216).—See XX.

Theobromine from cacao waste (G.P. 423,761).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Error introduced by the presence of bismuth in the colorimetric determination of iron in red lead. Colorimetric determination of bismuth. H. HEINRICHS and M. HERTRICH (Glastech.

Ber., 1924, **2**, 112—115 ; Chem. Abstr., 1926, **20**, 561).—Bismuth should be removed as sulphide before colorimetric determination of the iron with thiocyanate, or 5—10 c.c. of concentrated hydrochloric acid for 10 g. of red lead should first be added. The bismuth may be determined by dissolving the sulphide in dilute nitric acid, precipitating with ammonia, redissolving the precipitate in dilute sulphuric acid, adding 50—100 c.c. of 20% ammonium thiocyanate solution, and comparing the yellow colour produced with that of a similar mixture containing standard bismuth solution (1 c.c.=0·0002 g. of bismuth trioxide). Using 100 c.c. of ammonium thiocyanate, 0·00005 g. of bismuth trioxide may be detected. A. A. ELDRIDGE.

Oxidation of metallic iron by a current of air in presence of iron salts. P. FIREMAN (Ind. Eng. Chem., 1926, **18**, 286—287).—Scrap iron was placed in a receiver within a large tank containing ferrous sulphate or chloride solution and air blown through the whole after the temperature had been raised to about 60°. Hydrated ferric oxide was rapidly formed, at first of a yellow colour, then deepening to brown and finally to red-brown or purple-brown. A sample of this red-brown pigment, after drying on the water bath, contained 13·6% of moisture, and a study of the dehydration curve suggests that it may be ferric oxide monohydrate. The value of *d*, corrected for excess moisture, was found to be 4·376, which is in fair agreement with that of natural monohydrate. C. IRWIN.

Cellulose nitrate lacquers. A. E. LAIN (J. Oil and Colour Chem. Assoc., 1926, **9**, 33—44).—The modern lacquers that are in demand for motor body finishes and other purposes that have hitherto been served by oil varnishes owe their superiority over the older metal lacquers to their relatively high content of solids. This has been made possible by the advent of "low viscosity cellulose nitrate." A lacquer containing 26% of solids (say 16% of cellulose nitrate and the remainder resins and plasticisers) can be sprayed and rubbed to a satisfactors finish. The advantages claimed for lacquer coatingy are rapid drying and hardening to a permanent film, resistance to water, petrol, etc., and absence of "bloom." Resins differ greatly in their power to impart adhesion, gloss, and rubbing qualities, and the author discusses the resins of commerce from these angles. He also reviews lacquer solvents, methods of manufacture, application and pigmenting of lacquers. S. S. WOOLF.

Need for research in the oil and colour industry. H. H. MORGAN (J. Oil and Colour Chem. Assoc., 1925, **8**, 255—268).—A general survey and analysis of various problems of the paint and varnish industry requiring investigation, including the following :—storage and keeping qualities of paints, enamels, and varnishes ; changes occurring during application, drying, and weathering ; properties of the raw materials, their influences on the dried film, and the changes which take place during manufacture ; structure and adhesive properties of the film ; polymerisation and drying of linseed and tung oils ; viscosity and plasticity of paint and varnish films ; effect of solvents ; methods of measuring fundamental properties of pigments (particle size and shape, oil absorption, staining power, opacity, and colour) and of dried films (gloss, opacity, hardness, flexibility, tensile strength, and water absorption), and relationship between these properties and behaviour in practice.

Leather japanning. LATTEY.—See XV.

PATENTS.

Titanium pigments. C. WEIZMANN and J. BLUMENFELD (E.P. 247,296, 12.7.22).—Colloidal titanium compounds with desired physical characteristics for various purposes are produced by the peptisation of precipitated titanium compounds ; *e.g.*, titanium oxide or hydroxide precipitated by hydrolysis, before being dried, is submitted to a dispersion process using small quantities of acid, alkali, salts of titanium, or any substance which diminishes the surface tension of the liquid phase. The product when dried forms clots which are easily reduced to powder ; this with linseed oil gives a paste containing ultra-microscopic particles. Films obtained by drying this paste are very adhesive and are permanent in colour. Fillers may be introduced into the titanium pigment, before, during or after the peptisation process. D. F. TWISS.

Carbon products [pigments]. H. H. WARD (E.P. 247,364, 22.1.25).—A mixture of dried ground peat and bone meal is heated in a closed retort at about 300° until carbonisation is complete. The charcoal is quenched while incandescent, dried, and mixed with gas carbon black and a small amount of ground soap powder. The whole, when ground to a fine powder, is suitable as a filler for rubber goods, and for making printing inks etc. S. S. WOOLF.

Manufacture of varnish. F. SCHWARTZ and E. GIL-CAMPORRO (Assee. of L. LEACH) (E.P. 231,457, 9.3.25. Conv., 26.3.24).—A varnish completely soluble in water, easy to apply, and imparting to objects coated therewith a brilliant, hard, resistant, elastic surface is prepared by saponifying gums or resins in the presence of ammonium "ricinate" (ammonium soaps of castor oil fatty acids) and then mixing the product with an oil saponified with ammonia, a certain amount of albumin, casein, or similar substance dissolved in water being also added. The ammonium "ricinate" is prepared by saponifying castor oil with caustic soda, decomposing the soap by means of hydrochloric acid, and treating the liberated fatty acids with aqueous ammonia The saponification of the gums and resins by ammonia is facilitated by the ammonium "ricinate." The following proportions may be used for preparing the ammonium soap of the resin : sandarach 100 kg., ammonium "ricinate" 15 litres, ammonia 30 litres, water 300 litres. For the final varnish mixing the

following proportions are given: soap of resin 100 pts., ammonium soaps 15 pts., water 100 pts., casein (30% ammoniacal solution) 15 pts.

H. M. LANGTON.

Resinous compositions suitable for building and analogous purposes. W. H. BOORNE and C. C. L. G. BUDDE (E.P. 247,620, 11.10.24).—By heating ordinary rosin with formaldehyde in the presence of a catalytic substance, *e.g.*, a metal oxide or hydroxide, such as calcium hydroxide, a reaction product is obtained which when dry and mixed with fillers, such as sawdust, asbestos, or sand, will yield "moulding powders." The primary resinous product is fusible when heated and at 120° gases and vapours are copiously evolved; the residual product does not melt when further heated and may likewise be made to give moulding powders by the addition of fillers.

D. F. TWISS.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Weber's dinitrocaoutchouc and a nitrosate $C_{10}H_{16}N_2O_6$. F. EMDEN (Kautschuk, Feb., 1926, 31—33).—Doubt is thrown on the existence of the dinitrocaoutchouc $C_{10}H_{16}N_2O_4$ recorded by Weber and other workers. The action of nitrogen tetroxide in carbon tetrachloride on a solution of purified rubber near 0° yields a product $C_{10}H_{16}N_2O_6$; this primary product, the formation of which must involve an oxidation process, is the same whether from *Hevea brasiliensis* or from guayule rubber; on keeping it appears to undergo intramolecular change being at first insoluble and later becoming soluble in acetone. D. F. TWISS.

Determination of quality of raw rubber. W. GREINER and J. BEHRE (Kautschuk, Feb., 1926, 29—31).—Investigation of the surface tension characteristics of rubber solutions by comparing the rate of rise of a solution of 0·5 g. in 100 c.c. of benzene in a strip of filter paper, shows that the rate of rise of the solvent is most reduced by pale crêpe rubber, the effect of smoked sheet rubber, plain sheet, *Ficus* crêpe, guayule, and native remilled crêpe being increasingly feebler in the order stated. If the height of the ascent is plotted against the logarithm of the time, the result is a straight line. Mastication of the rubber increases the rate of ascent.

D. F. TWISS.

Stress-strain relationship of rubber under compression. C. H. BIRKETT (Trans. Inst. Rubber Ind., 1926, 1, 368—374).—Progressive vulcanisation causes an alteration in the compression-load curve for rubber similar to that observed in the ordinary stress-strain curve; the alteration in "slope" is negligible. The compression-load curve shows also other analogies with the stress-strain curve, but the initial inflexion is absent. D. F. TWISS.

Absorption of water by rubber. C. R. BOGGS and J. T. BLAKE (Ind. Eng. Chem., 1926, 18 224—232).—The absorption of water by rubber is analogous to the swelling of rubber in rubber solvents and of other colloids in water. There is no change in total volume and the rate of absorption is not appreciably affected by pressure although increased by rise of temperature. The rate of absorption follows no simple mathematical law. Substances dissolved in the water may have a marked effect on the absorption, salt, sugar, glycerol and, in particular, sodium hydroxide, decreasing the rate whereas ammonia, aniline, and acetic acid increase it. No great increase in the resistance to water resulted from the use of other coagulants than acetic acid or the presence of additional chemicals during coagulation. The resistance of pale crêpe rubber to water increases with age or with progressive vulcanisation; smoked sheet and Para rubber are comparable with crêpe, but "sprayed" rubber is exceptionally absorptive. The presence of water-soluble constituents increases the absorptive capacity of rubber and the washing of rubber increases its resistance to water. The absorption of water is due to the non-rubber constituents and the removal of the resinous, protein and other ingredients yields rubber with a very low absorption. Gutta-percha is somewhat more resistant than pale crêpe rubber to water, but less so than purified rubber.

D. F. TWISS.

Plasticity of unvulcanised rubber. R. W. GRIFFITHS (Trans. Inst. Rubber Ind., 1926, 1, 308—341).—A review of the methods for the measurement of the degree of plasticity of rubber. The "recovery" of rubber from the effect of milling is more marked above 55° than below and to prevent hardening from this cause rubber should be cooled as rapidly as possible after mastication. A formula is deduced indicating the relation between the results obtained with the parallel plate plastometer (Williams, B., 1924, 480) and the magnitude of the absolute viscosity. D. F. TWISS.

Blending of compounding ingredients [for rubber]. T. R. DAWSON (Trans. Inst. Rubber Ind., 1926, 1, 359—367).—The results of Wiegand (B., 1925, 816) as to the reinforcing action of various ingredients were affected by the presence of litharge in his parent mixing; with rubber and sulphur without litharge, whiting can exert distinct reinforcing effect. This is a special case of the general absence of simple additive relationship in the joint effect of various rubber ingredients.

D. F. TWISS.

Low-temperature vulcanisation [of rubber]. F. THOMAS (Trans. Inst. Rubber Ind., 1926, 1, 375—386).—Mainly a review of the activation and application of ultra-accelerators. Litharge activates the dithiocarbamate and thiuram disulphide accelerators, although to a less extent than zinc oxide; towards the zinc alkylxanthates litharge has a retarding effect. Antimony sulphide impairs the activity of certain organic accelerators, *e.g.*, the xanthates, and its colour is often affected. Carbon black also exerts a retarding influence and so tends to check the pre-vulcanisation of ultra-accelerated mixtures. D. F. TWISS.

Acceleration of vulcanisation [of rubber] by alkaloids. B. J. Eaton and R. O. Bishop (Malay. Agric. J., 1926, 14, 8—12).—Small quantities (0·01—0·1%) of emetine exert a distinct accelerating effect but nicotine, cocaine and hyoscine in similar proportion are ineffective. Powdered ipecacuanha root and root bark also accelerate vulcanisation; the residues from the extraction of emetine may therefore be of value in this direction.

D. F. Twiss.

Patents.

Manufacture of rubber. L. le W. Hamon (E.P. 247,712, 5.1.25).—Rubber (100 pts.) is compounded with carbonised peat (25—35 pts.); the product is adapted for use in tyres and floor coverings.

D. F. Twiss.

Age-resisting rubber compound. E. W. Fuller, Assr. to Fisk Rubber Co. (U.S.P. 1,573,928, 23.2.26. Appl., 27.4.25).—Products with improved ageing properties are obtained by adding an alkali or alkaline-earth salt of α-nitroso-β-naphthol to the rubber mixture before vulcanisation.

D. F. Twiss.

Carbon pigments (E.P. 247,364).—See XIII.

XV.—LEATHER; GLUE.

South American quebracho extracts. W. Vogel (Collegium, 1926, 66—73).—The quebracho growing area is about 675,000 sq. km. included in latitudes 21—30 and longitudes 57—64. For the manufacture of quebracho extracts only red quebracho (Span., *Quebracho colorado*), the heartwood of *Quebracho Lorentzii* and *Schinopsis Balansae* is used; white quebracho (*Aspidosperma Quebracho*) contains scarcely any tannin. Distinction is also made between quebracho from different districts, quebracho Chaqueno and quebracho Santiagueno. The quebracho Chaqueno ("chaco") is preferred. The composition of the heartwood, sapwood, and bark respectively of *Quebracho colorado*, Chaqueno, by the filter-bell method of analysis is: tans 20, 2·2, 4·5; non-tans 1·5, 2·0, 1·7; insoluble matter 59·5, 53·3, 81·4; water 19, 42·5, 12·4%. The quebracho logs are sawn into thin boards, rasped, leached with hot water in wooden or copper vats, the liquor is settled and concentrated to *d* 1·165—1·210 in double- or triple-effect evaporators. Cold-soluble extracts are prepared by treating the ordinary extract with a mixture of sodium sulphite and bisulphite at 90—95° until it is entirely soluble in cold water. The yield of extract is 27—28% of crude and 30—31% of sulphited. The composition of crude and sulphited extracts respectively by the shake method is: tans 63%, 66·5%, non-tans 8%, 11·5%, insoluble matter 7%, 0%, water 22%, 22%. Sulphited extracts penetrate and tan the pelt quickly, but do not give weight. The nature and degree of sulphiting have an important bearing on the quality of the extract. The non-tans are greatly affected by the quality of the wood used, the temperature of the leach liquors, and the amount and composition of the water used in leaching. The higher the temperature and the greater the amount of water, the greater the amount of non-tans in the extract. The insoluble matter in crude extracts varies from 3 to 11%, and consists chiefly of phlobaphens; it possesses tanning properties, and is useful in drum tanning, especially at higher temperatures, and in combination with other tanning materials, when a large portion enters the leather and imparts weight and solidity. The moisture content should be determined on a sample taken from the inside of the package. It is usually 10—12%. All analytical results should be calculated to the same percentage water content, viz., 22%. The insoluble matter should be determined for practical purposes by making solutions of *d* 1·014, 1·029, and 1·045, respectively. These solutions should be clear for sulphited extracts and remain clear for 1 day. The total annual production is 222,000 tons in Argentina and 46,000 tons in Paraguay. The annual consumption of wood is 1,300,000 tons. There are reserves of 55,000,000 tons which will last 40 years at the present rate of consumption.

D. Woodroffe.

Insoluble matter in oakwood extract. P. Pavlovitsch (Collegium, 1926, 78—81).—In experiments on solutions of oakwood extract of different strengths, the insoluble matter was taken as the difference between the total solids determined immediately after making up and again after allowing to settle for 8 hrs. at 15°. The insoluble matter increased with increase in solution strength up to *d* 1·064 and diminished above that strength. A rise of temperature from 20° to 40° reduced the amount of insoluble matter by half. The determination of insoluble matter by filtering through filter paper gives 30% higher figures owing to adsorption by the paper. Oakwood extracts should be clarified at a strength of *d* 1·064 by cooling, then adding 1 pt. of ox blood per 1000 pts. of liquor, or lead acetate, alum and bisulphite, or other coagulants. One of the best coagulants is cellulose (1 pt. to 15,000 pts. of liquor) which has previously been converted into a paste by boiling with hydrochloric acid (1 pt. HCl to 30 pts. of cellulose).

D. Woodroffe.

Synthetic tanning materials. E. Wolesensky (Tech. Papers, U.S. Bur. Standards, 1925, 20, [302], 1—45).—A preliminary study has been made, chiefly qualitative, of the methods of preparation and of the tanning properties of a number of synthetic materials prepared by condensation of sulphonated products of benzene, toluene, naphthalene, phenol, cresol, and α- and β-naphthols with formaldehyde (and in a few cases also with acetaldehyde and furfuraldehyde), and of naphthalenesulphonic acid with naphthalene, phenolsulphonic acid, and glycollic acid respectively, together with a few non-sulphonated condensation products of resorcinol and pyrogallol with the same aldehydes. Most of the products studied possess tanning properties. The simple sulphonic acids of the aromatic hydrocarbons or the corresponding phenols possess no tanning properties.

Sulphonated condensation products derived from the hydrocarbons were generally unsatisfactory as tanning agents. The resins obtained by condensing naphthalene or toluene with formaldehyde were found to be too difficult of sulphonation by direct treatment with sulphuric acid, and the condensation products of the sulphonic acids of toluene and naphthalene with formaldehyde had poor tanning properties. The products obtained by condensing the sulphonic acids of the monohydric phenols with formaldehyde possessed true tanning properties and produced light weighing, soft, pliable and strong leathers. The condensation can be carried out in dilute solutions at higher temperatures, or in concentrated solutions with or without cooling. The products from the condensation of phenol- or cresol-sulphonic acids with formaldehyde in dilute hot solutions give a nearly white leather, but condensation in concentrated solutions gives a coloured synthetic tannin which darkens the leather. Synthetic tannins from naphtholsulphonic acids produce dark coloured leathers. Resins from phenol or cresol and formaldehyde, if not too highly condensed, can be readily sulphonated by means of strong sulphuric acid, but more care is required than in the sulphonation of the original phenol or cresol. These sulphonated resins are good tanning agents and possess better filling properties than the products mentioned above. Formaldehyde was replaced by acetaldehyde in a sulphonated phenol–aldehyde resin and the product had good tanning properties and better filling properties than any other product, but the yield of condensation product was low. The condensation products obtained from resorcinol or pyrogallol and formaldehyde or acetaldehyde possessed greater filling properties than any of the sulphonated products and are capable of producing a very good quality of leather for some purposes. Furfuraldehyde reacts less readily but under proper conditions will also yield satisfactory results.

D. WOODROFFE.

Chrome tanning. III. Tanning effect of some complex chromium compounds. E. STIASNY and L. SZEGÖ (Collegium, 1926, 41—65; cf. B., 1925, 139, 559, 560).—A chromium sulphate solution of 33·3% basicity was mixed with sodium sulphite in amounts ranging from 0·25 to 10 mols. of sulphite per 1 mol. $Cr(OH)SO_4$. The solutions were diluted to a strength of 2·5 g./litre Cr, and kept for 24 hrs. before testing. Additions of sodium sulphite above 0·5 mol. prevented precipitation of the chrome liquor by weak solutions of ammonia. The resistance to precipitation was lost if the excess sulphite was removed. The sulphite–chromium complex was therefore stable only in presence of excess sodium sulphite. With small additions of sodium sulphite, the chromium migrated to the cathode, but to the anode with 1 mol. of sulphite or more. The p_H value increased from 2·7 with no sulphite, to 8·1 with 10 mols. The maximum amount of SO_3 combined with 1 mol. Cr was 0·47 mol. and the compound formed is probably $[Cr_2(OH)_2(SO_3)(SO_4)_2]''Na_2$. The liquor containing this compound, on keeping, yields a precipitate which contains no sulphate and has 3 Cr atoms to 2 SO_3 radicals. The tanning properties of the liquor increase considerably with additions of sulphite up to 1½ mols. and decrease rapidly with further additions. Gustavson (B., 1923, 65) obtained similar results with sodium acetate and formate, as also did Burton (B., 1924, 880). The tanning velocity—which has usually been considered as a measure of astringency—increases with additions of sodium sulphite although the solution contains chromium in the anion, and it is concluded that the laws which govern the ordinary basic chromium sulphates do not apply to these chromium complexes which manifest anodic migration. With ordinary basic chromium sulphates, the velocity of tannage and the amount of chromium absorbed increase as the precipitation number diminishes. The proportion of chromium combined with hydroxyl groups is not the important factor in these chromium complexes because the increase of the negative radicals combined with the chromium still more increases their negative charge and their resistance to alkali. The results show there is no connexion between the ionic migration of the chromium complexes and the tanning properties. Experiments with sodium oxalate give similar results to those obtained with sodium sulphite. The behaviour of "permutit" is not always analogous to that of hide, as Gustavson claims.

D. WOODROFFE.

Sulphur tannage. A. W. THOMAS (Ind. Eng. Chem., 1926, 18, 259—260).—Sulphur hydrosol was prepared by the slow addition of 3*N*-sodium thiosulphate to 17*M*-sulphuric acid in an ice bath. The colloidal sulphur was precipitated by sodium chloride, and repeatedly redissolved and reprecipitated until a clear yellow solution of colloidal sulphur was obtained. Portions of hide powder were shaken with the sulphur hydrosol, then washed with a large volume of water, dehydrated, dried, and weighed. The increase in weight was due to sulphur and was very small, 0·191% or less. Practically all the sulphur was extractable by carbon disulphide. The sulphur fixation was slow and increased with the amount of colloidal sulphur present. Colloidal sulphur does not precipitate gelatin. Acidified hide powder fixed more sulphur. A decrease in acidity was accompanied by a decrease in the fixation of the sulphur, which was in accordance with the Procter–Wilson theory of tanning. The sulphur-treated hide powder was more resistant to the action of cold water than raw hide substance, but readily decomposed in hot water. The combination of the colloidal particles and the collagen apparently does not result from a mere deposition of the sulphur owing to the coagulation of the sulphur hydrosol. It is due to the combination of the collagen cations and the anion, xS,yS_5O_6H' of the complex $xS, yS_5O_6H_2$, which forms the disperse phase of the hydrosol. The results are contrary to Apostolo's (cf. B., 1913, 877). D. WOODROFFE.

Effect of splitting on the tensile strength of leather. J. A. WILSON and E. J. KERN (Ind. Eng.

Chem., 1926, **18**, 312—313).—Rectangular pieces of leather were cut on either side of, and 10 cm. from, the backbone of each of a number of representative skins of finished vegetable- and chrome-tanned calfskins. The pieces were cut into 25 strips of equal size, numbered consecutively, and the odd-numbered pieces tested for tensile strength. The even-numbered pieces were split into two layers. The thickness varied with the different numbers. The tensile strength of each split was measured and compared with the calculated strength of the unsplit strips. The results show that the tensile strength is not uniform throughout the thickness, the grain layer being relatively weak. The strength of the leather is in the reticular layer ("flesh"). Splitting always causes a loss in strength per unit width and the sum of the strengths of the two splits is always less than the strength of the unsplit strip. Chrome leather loses 60% of its total strength, whereas vegetable-tanned leather loses 48% only. Cutting away the grain layer to a depth less than 48% for vegetable-tanned leather or 22% for chrome leather, increases the strength per unit cross section of the remaining flesh layer. D. WOODROFFE.

Leather japanning. W. T. LATTEY (J. Oil and Colour Chem. Assoc., 1926, **9**, 44—49).—The leather finish is comprised of filler coats, colour coats, and varnish coats, the hardness increasing progressively from the primer to the finishing coat, whereas the reverse is the case in the painting of timber and metal, when the finishing coat is the most elastic. Linseed oil heated with iron and aluminium driers for 4—5 hrs. at 300° gives a stiff jelly which is spread in a thick coat on the leather (previously rendered supple by the rubbing in of not more than 10% of a semi-drying oil). Further priming coats of a like nature are applied, each coat being dried for 24 hrs. at 70°, and the tackiness that is apparently inevitable at this stage is removed by exposure to direct sunlight. An alternative to the stoving process is the use of sunlight throughout, but this is dependent on the weather in general, the temperature (which must be relatively low to keep the linseed jelly stiff enough to prevent it from sinking in), and the relative humidity. The last factor is of importance in connexion with the swelling of the fibres, on which depends the elasticity of the skin. A qualified success only is attained by the use of artificial light to replace sunlight. For the subsequent coats almost any good quality paint or varnish may be used. Cellulose nitrate compositions also are now used in leather treatment. S. S. WOOLF.

See also A., April, 351, **Influence of hydrogen-ion concentration on viscosity and elasticity of gelatin solutions** (FREUNDLICH and NEUKIRCHER). 409, **Tannin in kino of** ***Eucalyptus calophylla*** (MCGOOKIN and HEILBRON).

Variations in absorptive power. EFFRONT.—See XVIII.

Physical method for examination of gelatin. THORNE BAKER and DAVIDSON.—See XXI.

PATENTS.

Depilating hides and skins. M. BERGMANN (E.P. 247,826, 6.7.25).—Hides and skins are treated first with inorganic and organic bases, *e.g.*, ammonia, ethylenediamine, piperidine, pyridine, or carbamides or compounds of these bases, especially the sulphides, or further with soluble silicates, separately or mixed, and afterwards treated with the ordinary depilating agents. For sheepskins a solution containing 10 litres of ammonium sulphide solution or 10 litres of 25% ammonia per cub. m., and for calf skins a solution containing 10 litres of ammonium sulphide solution and 10 litres of commercial water-glass per cub. m. are recommended. D. WOODROFFE.

Extraction of glue and fatty matters from bones and the like. C. H. SHEARMAN (E.P. 247,347, 6.1.25).—Bones and the like are extracted with hot water in a vertical cylinder furnished with a charging and discharging orifice at its upper and lower ends respectively, each orifice being fitted with a steam- and water-tight lid. A perforated inverted cone extends from the lower orifice about halfway up the cylinder to facilitate discharge of the solid contents. One or more external pipes fitted with injectors connect the lower end of the cylinder with a horizontal perforated ring within the upper part to maintain circulation of the liquor. Discharge pipes are arranged near the top and bottom of the cylinder, and means provided for heating and injecting steam into the cylinder. A colloid coagulant, *e.g.*, $\frac{1}{2}$—1% of alum, is added to the water. After simmering with hot water for 4 hrs. water or weak glue liquor is introduced into the bottom of the cylinder and the fatty matter and strong glue liquor are discharged at the top. The cylinder is closed and the contents are subjected to a steam pressure of 5 lb. per sq. in. for 2 hrs., the liquor is discharged as before and, the cycle of operations is repeated at pressures of 10, 15, 20, 25, 30, and 35 lb. per sq. in. A series of cylindrical vessels may be used and weak liquors obtained at high pressures used on fresh material. D. WOODROFFE.

XVI.—AGRICULTURE.

Distribution of ***Azotobacter*** **in Bavarian soils with reference to the reaction of the soils and the content of calcium carbonate and phosphoric acid.** H. NIKLAS, H. POSCHENRIEDER, and A. HOCK (Zentr. Bakt. Parasitenk., Abt. II, 1925, **66**, 16—28; Chem. Zentr., 1926, I, 1876).—The relation between the p_H of the soil and the distribution of *Azotobacter* differs according to whether the extract is made with water or with potassium chloride solution. With water, no development is found at a p_H less than 5·6 and growth is best at a p_H greater than 6·5; the corresponding figures with potassium chloride solution are 4·5 and 6·0. A high calcium content favours development; grass land conditions are less suitable than arable land. *Azotobacter* may occur in soils deficient in phosphoric acid but, even with favourable reaction and high calcium content, does

not develop or fix nitrogen. In some soils it fails to develop even when the conditions in regard to reaction, calcium, and phosphoric acid are suitable.
C. T. GIMINGHAM.

Determination of the potassium and phosphoric acid requirements of soil from the molecular composition according to Ganssen. HUNNIUS (Landw. Jahrb. Schweiz, 1926, **63**, 145—156; Chem. Zentr., 1926, I, 1698).—The manurial requirements of the soil calculated by the method of Ganssen from the molecular composition of the aluminium silicates extracted from the soil by boiling concentrated hydrochloric acid do not correspond to the requirements determined in field experiments and by the methods of Mitscherlich and Neubauer. A better indication is obtained by the total content of nutritive elements and the proportion of colloidal silica. As the molecular composition is not directly related to the soil reaction, the degree of saturation of the soluble aluminium silicates is not an exact measure of the exchange acidity. B. W. CLARKE.

Determination of manurial requirements of soils by laboratory methods. (1) DENSCH (Z. Pflanz. Düng., 1926, **B5**, 97—104; cf. B., 1925, 858). —Critical experiments on Neubauer's seedling method for determining the phosphoric acid and potassium requirements of soils are recorded. The amount of phosphoric acid or potassium per 100 g. of soil as determined by this method was compared with the actual response of the soil to these nutrients in field or pot experiments with a variety of cultivated plants. The results were irregular and there was no general agreement between the two methods. Further, the figures obtained by Neubauer's method on the same soils in different laboratories were, in many cases, widely divergent. Possible variations in technique, which may account for such divergencies, are discussed. It is considered that further work on the method is required in order to define more precisely the optimum conditions.

(2). O. LEMMERMANN (*ibid.*, 105—117).—Neubauer's method is discussed. The assumptions involved in the calculation of the limiting values per 100 g. of soil, which are taken to indicate lack or sufficiency of phosphorus and potassium, are criticised, and numerous experiments are described in which the phosphoric acid requirements of soils determined by the Neubauer and by the citrate method are compared with the results obtained in field and pot experiments. The three methods are in general agreement in many cases but by no means in all. The Neubauer method needs considerable refinement before it can give results of certain value.

(3). E. BLANCK (*ibid.*, 118—125).—Experiments on the use of various acid solvents for determining "available" nutrients in soil are described (cf. B., 1926, 70). The relative solubility of the phosphoric acid of soils in 0·5% citric acid is in agreement, in a number of instances, with the response of the soil to phosphate manuring. Results obtained by Neubauer's method agree in some cases but not in others.

(4). H. NEUBAUER (*ibid.*, 126—129; cf. B., 1925, 220).—A reply to the criticisms of the author's seedling method contained in the papers by Densch and Lemmermann referred to above.

(5). E. A. MITSCHERLICH (*ibid.*, 132).—Some criticisms of Neubauer's method from the standpoint of plant physiology.

(6). DENSCH (7). O. LEMMERMANN (*ibid.*, 130—131, 133—143).—Further discussion of Neubauer's method and comparison of the results with those obtained by other methods. C. T. GIMINGHAM.

Carbon dioxide and plant yield. A. RIPPEL (Z. Pflanz. Düng., 1926, **B5**, 49—64).—A crictical discussion of recent work bearing on the possibility of increasing crop yields by artificially regulating the amount of carbon dioxide in the atmosphere. It is considered that the raising of this question has added to our knowledge from the point of view of plant physiology but that it is of little or no practical importance as regards plant growth in the field.
C. T. GIMINGHAM.

Carbon dioxide manuring. GERLACH (Z. Pflanz. Düng., 1926, **B5**, 65—69; cf. B., 1925, 684).—In field experiments, slightly lower yields of yellow lupins and of potatoes were obtained on plots where the air was enriched by additional carbon dioxide during the growing period than on plots not so treated. A comparison of stable manure with artificial fertilisers gave results with potatoes in favour of the former, but reasons are given why this cannot be explained as due to an increased content of carbon dioxide in the air. C. T. GIMINGHAM.

Importance of soil carbon dioxide for the nutrition of plants and the action of some humus or carbon dioxide manures. O. LEMMERMANN (Z. Pflanz. Düng., 1926, **B5**, 70—84; cf. B., 1925, 110, 182).—Vegetation and field experiments show that soil receiving dung and green manure in addition to mineral fertilisers produced about 35% more carbon dioxide than the same soil with minerals only. In spite of this, the average percentage of carbon dioxide in the air over the dunged plots was no higher than over the undunged plots. The increased yields obtained with organic manures are not thought to be due to carbon dioxide. Several so-called "humus" or "carbon dioxide" manures were tested on potatoes with inconclusive results. C. T. GIMINGHAM.

Carbon dioxide [and plant growth]. P. EHRENBERG (Z. Pflanz. Düng., 1926, **B5**, 85—86).—Experiments are referred to indicating that, at all events on highly cultivated soils, additional carbon dioxide is ineffective for promoting plant growth.
C. T. GIMINGHAM.

Effect of tar and tar vapours on the soil. EWERT (Landw. Jahrb. Schweiz, 1926, **63**, 103—127; Chem. Zentr., 1926, I., 1698).—The lower-boiling constituents of tar are harmful to plant roots and to bacteria in the soil; radishes are especially sensitive to the influence of tar. Where the air contains a relatively high proportion of tarry vapours, *e.g.*, in the neighbourhood of tar distilleries, the amount of

tar absorbed by the soil is insufficient to produce a harmful effect, although the vapours themselves are very harmful to the parts of the plant above the ground. Tar therefore does not act as a soil poison in the same way that silica does. Very small quantities of tar vapour in the air, however, are sufficient to poison the leaves. B. W. Clarke.

Fundamental investigations on basic slags. McArthur (Scottish J. Agr., 1925, 8, 72—73; Chem. Abstr., 1926, 20, 470).—Under particular soil conditions the dicalcium silicate present in open-hearth, fluorspar basic slag can function as an ingredient for the correction of soil acidity. The molecular composition of a slag is of greater significance than its percentage composition. A. A. Eldridge.

Experiments with calcium nitrate. E. Blanck and A. Hahne (J. Landw., 1926, 74, 51—64).—Pot experiments with oats and field trials with sugar beet show little difference between the efficiency of sodium nitrate and of a proprietary brand of calcium nitrate ("B.A.S.F. Kalksalpeter") as sources of nitrogen. Preliminary laboratory experiments indicate that the effect of the calcium nitrate on the physical properties of soil was not unfavourable. C. T. Gimingham.

Surophosphate or Dasag manure. E. Blanck and F. Alten (J. Landw., 1926, 74, 39—49).—Dasag manure (Surophosphate or Surogatphosphate) is prepared from sewage and all kinds of town refuse by addition of peat and sulphuric acid; it contains about 0·7% N, 1·0% K_2O, and 4·0% P_2O_5. Pot experiments on several soils show that this material has little or no effect on the growth of maize even when used in amounts much greater than could be employed in practice. C. T. Gimingham.

Utilisation of disinfectants containing mercury in disinfecting baths [for seeds]. G. Hilgendorff (Z. angew. Chem., 1926, 39, 377—379).—The loss in disinfecting power, *i.e.*, diminution in mercury content, of solutions of various disinfectants (Uspulun, Germisan, Urania disinfectant) used for treating seeds, cereals, etc., was determined, the results differing somewhat from those obtained by Krauss (B., 1926, 30) under slightly different conditions. The absorption of the disinfectant by the seeds and the effect of the extractive matter (carbohydrates, proteins, etc.) which collect in the bath, are the most important factors influencing the change in mercury concentration of the solution. Temperature has only a slight effect. No comparative figures are yet available for large-scale operations as distinguished from laboratory-scale experiments. B. W. Clarke.

Soil core sampler. E. B. Powell (Soil Sci., 1926, 21, 53—57).

Effects of various methods of applying fertilisers on crops and on certain soil conditions. D. G. Coe (Soil Sci., 1926, 21, 7—21, 127—141).

See also A., April, 347, **Quantitative adsorption analysis by Wislicenus' method** (Lorenz). 352, **Relation between properties and chemical composition of soil colloids** (Anderson and Mattson). 354, **Coagulation of clay** (Gallay). 358, **Equilibrium in system arsenic pentoxide–barium oxide–water** (Hendricks). 377, **Wiegner's elutriation apparatus** (Gessner). 438 **Manganese and plant growth** (McHargue); **Physical and chemical factors in growth of asparagus** (Working).

Patents.

Manufacture of nitrogenous fertilisers from calcium cyanamide. Chem. Fabr. Heppes & Co., and J. B. Carpzow (G.P. 422,623, 7.7.23).—Calcium cyanamide mixed with sewage sludge, sapropel, saprocoll, etc., is dried and granulated. A non-corrosive, non-dusting product is obtained, which is not washed away from the soil by rain water. B. W. Clarke.

Production of urea. Norsk Hydro-Elektrisk Kvaelstofaktieselskab (Nor. P. 39,744, 13.11.22).—Urea is obtained by passing carbon dioxide at 80—160° (preferably at 130—140°) under pressure, over compounds of ammonia with salts such as ammonium chloride, calcium chloride, zinc chloride, aluminium chloride, or calcium nitrate. The product is used direct as a fertiliser, or is worked up into urea salts. L. A. Coles.

Insecticidal composition. C. N. Hand, Assr. to Rubber Service Laboratories Co. (U.S.P. 1,573,490, 16.2.26. Appl., 25.3.25).—A dusting composition, comprising a thiourea, is claimed for destroying insects. C. T. Gimingham.

Improving the adhesion of spray powders to plants. Farbenfabr. vorm. F. Bayer & Co. (G.P. 412,516, 26.6.21).—The powder, *e.g.*, sulphur, is mixed or saturated with a powdered alkali soap or soap solution and powdered lime or magnesia added. This prevents the washing away of the insecticide by dew or by rain, owing to the formation of a calcium soap which fixes the sprayed powder on the plant. The process may be applied to pumice stone, nicotine, Paris green, copper sulphate, etc., in the powdered form. B. W. Clarke.

Production of hydrocyanic acid for use as an insecticide. Chem. Fabr. H. Stoltzenberg (G.P. 420,729, 21.10.24).—Hydrogen cyanide is produced by heating the salts of organic bases such as trimethylamine, betaine, hexamethylenetetramine, etc. (preferably the hydrochlorides). The salts are less hygroscopic than the free bases and a higher yield of hydrogen cyanide is obtained, *e.g.*, 70—80% with betaine hydrochloride compared with 50—60% with free betaine. B. W. Clarke.

Manufacture of solid calcium nitrates. J. Y. Johnson. From Badische Anilin & Soda-Fabr. (E.P. 249,370, 29.5.25).—See U.S.P. 1,564,410; B., 1926, 139.

Removing silica from acid ore leaches (U.S.P. 1,571,054).—See X.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Purification of sugar solutions. K. URBAN, Assr. to S. ELBOGEN (U.S.P. 1,577,389, 16.3.26. Appl., 2.6.24).—See E.P. 240,253; B., 1925, 1005.

Electro-osmotic purification of saccharine juices. K. WOLF and E. LANGSTEIN, Assrs. to AMER. ELECTRO-OSMOSIS CORP. (U.S.P. 1,577,669, 23.3.26. Appl., 29.3.24).—See E.P. 214,578; B., 1924, 921.

Feeding stuffs (Swiss P. 95,512).—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Stream-line filter and its applications to brewing and bottling. H. L. HIND and J. A. PICKARD (J. Inst. Brew., 1926, 32, 97—102).—Beer for bottling can be dealt with by the stream-line filter (Hele-Shaw, J.S.C.I., 1923, 353 T). Protein hazes are removed without loss of colour of the beer or removal of colloids associated with body, flavour, and head retention, and the beer, so filtered, is rendered sterile and remains bright for almost any length of time. Arrangements can be made for the liquid undergoing filtration to remain under a positive pressure of carbon dioxide. The quantity of beer left in the filter is reduced to a minimum and is less than with pulp filtration. With slight modification the filter can be applied, more efficiently than the ordinary filter-press, to the separation of yeast from beer, with the advantages of cleaner working, elimination of filter cloth troubles, greater ease of sterilisation, lower cost of running, and fewer renewals. It is easily cleaned and can be sterilised without damage to the paper by passage of live steam. C. RANKEN.

Variations in the absorptive properties of yeast. J. EFFRONT (Compt. rend. Soc. Biol., 1925, 93, 1248—1250; Chem. Zentr., 1926, I., 2013—2014).—The properties of yeast depend upon its method of nutrition. A modified fermentation process in which carbon dioxide in the usual quantities, but no alcohol is produced, is brought about by adding yeast to a very dilute nutrient solution, and gradually adding the nutrient so as to maintain it at constant concentration. Under these conditions, the "vegetative" function of the yeast prevails, whereas in the usual method of fermentation, the action of the zymase is predominant. The two varieties of yeast can be separated by the difference between their absorptive capacity for sodium hydroxide and hydrochloric acid, preferably the latter, as they both absorb sodium hydroxide, whereas only the usual variety absorbs acids. On the addition of 12 to 41 c.c. of $0 \cdot 1N$-acid per 100 g. of dry yeast, the solution separates into two layers. Variations in the method of nutrition indicate that the cell membrane is impervious to hydrochloric acid. L. A. COLES.

Variations in absorptive power. J. EFFRONT (Monit. Sci., 1926, 16, 3—11, 29—36; cf. B., 1923, 67 A, 287 A; 1924, 763; 1925, 144).—A review of our knowledge on the subject of absorption especially by organised substances. The action of invertase is explainable by the assumption of a specific absorbent capable of fixing both sugars and hydrogen-ions so as to create a medium favourable to hydrolysis, while the products enter into solution and the catalyst once again becomes available. Vegetable materials differ widely in their water-absorbing powers. 1 g. of elder pith absorbs 60—80 g. of water, while vegetables and fruit pulps also absorb considerable quantities. Gelatin absorbs a maximum proportion of water at p_H 3·2, the amount diminishing as the p_H increases. At the isoelectric point absorption is relatively small. Further, the amount absorbed is less in the presence of alkali than of acid, but the p_H of the gelatin grains is more important than that of the medium. The consistence of yeast depends more upon the state of the contained water than on its amount, that is the relative proportions absorbed by the cells and that held between them. Vegetable juices appear to contain a substance which retards the action of diastase and of which the physical state is altered on warming, whereby it is retained on a filter, so that filtration will frequently increase the activity. Although yeasts absorb colouring matter from caramelised worts, when cultivated under certain conditions in the presence of calcium chloride this power is lost, probably owing to the selective development of those cells having no power of absorption. A modification of the absorptive powers of living cells may eventually explain the specific action of certain serums. It has been shown that diastase can change its function of analysis to that of synthesis and the formation of anti-bodies appears analogous. The augmentation of the specific toxicity of an antiseptic may well be the result of an increase in its power of absorbing a given parasite. D. G. HEWER.

Effect of sun on the ripening of the grape and the composition of the wine. E. HUGUES (Ann. Falsif., 1926, 19, 40—43).—The must from grapes damaged by over-exposure to sun, and characterised by their small size and red colour, and that from undamaged grapes, contained, in the first case, 115 g. of sugar, as dextrose, per litre, and in the second, 164 g., and the total acidity per litre as tartaric acid was 11·25 g. and 9 g. respectively. A smaller total quantity of wine was produced from the damaged grapes (6 litres less from 100 kg.), but the weight of marc was greater. Analysis of the two wines showed the first (from the damaged grapes) to be about 3 deg. inferior in alcohol content, of a poorer colour, but of higher acidity. D. G. HEWER.

Tannins for use in wine making. F. LEVALLOIS (Ann. Falsif., 1926, 19, 15—28).—It is suggested that definitions of the various types of tannins used in wine making in France should be formulated, together with some analytical limits, particularly

for tannin content, to prevent the widespread adulteration now prevailing. Methods for the differentiation and determination of tannin are critically reviewed. D. G. Hewer.

Dickinson alcohometer. E. A. Vuilleumier (Ind. Eng. Chem., 1926, 18, 261).—The small portable instrument consists essentially of a still and condenser to condense the alcohol in a sample to a fractional volume (1 c.c. is sufficient) and a graduated cylinder. Heat is supplied by a candle, and a hollow glass bead of definite density is put into the distillate, and water added until the bead floats. The density of the alcohol is then determined from tables. D. G. Hewer.

Detection of methanol [methyl alcohol] in alcoholic beverages. F. R. Georgia and R. Morales (Ind. Eng. Chem., 1926, 18, 304—306).—A modification of the Chapin method (B., 1921, 712 A) is recommended. Five c.c. of the alcoholic solution, previously diluted to 5% by volume of alcohol, are oxidised for 10 min. with 2 c.c. of a solution containing in 100 c.c. 3 g. of potassium permanganate and 15 c.c. of 85% phosphoric acid. The excess of permanganate is destroyed by 2 c.c. of a solution of 5 g. of oxalic acid in 100 c.c. of 1:1 sulphuric acid, and 5 c.c. of modified Schiff's reagent are immediately added, and after mixing the solution is kept for 10 min. for production of the characteristic colour due to formaldehyde. (Modified Schiff's reagent: 0·2 g. of rosaniline hydrochloride is dissolved in 130 c.c. of hot water, the solution cooled, treated with 2 g. of anhydrous sodium sulphite in 20 c.c. of water and 2 c.c. of concentrated hydrochloric acid, diluted to 200 c.c., and stored in well filled glass-stoppered amber bottles.) It is necessary to remove interfering substances: formaldehyde by pyrogallol, amyl alcohol, essential oils (except oil of angelica), fluid extracts of arnica and of ipecacuanha by a modified method of Thorpe and Holmes by shaking petroleum spirit with the 5% by volume distillate to which sodium chloride solution has been added; cinchonine, glycerol, liquorice, methyl-violet, quinine sulphate, resorcinol, strychnine sulphate, and tannic acid by distillation. D. G. Hewer.

Yeast preparation. Stern and Becker.—See XIX.

Patents.

Preserving yeast. L. Lindemann, Assr. to T. P. Hodge (U.S.P. 1,574,494, 23.2.26. Appl., 3.8.23).—The yeast is washed in water at 33—43° until the glycogen is completely or almost completely removed. D. G. Hewer.

Yeast preparation. R. Willstätter and H. Sobotka (U.S.P. 1,574,776, 2.3.26. Appl., 16.1.25).—A product rich in vitamins and enzymes, and suitable for use in baking and brewing, is obtained by mixing yeast with a disaccharide other than sucrose, in quantity sufficient to induce plasmolysis and to cause liquefaction but not to cause fermentation, *e.g.*, at least one-third of the weight of the yeast, plasmolysis being completed by heating the mixture. L. A. Coles.

Pasteurising liquids [beer]. F. Lassen (E.P. 249,377, 23.6.25).

Feeding stuffs (Swiss P. 95,512).—See XIX.

Oxalic acid from wood (G.P. 419,911).—See XX.

XIX.—FOODS.

Effects of fine grinding upon flour. C. L. Alsberg and E. P. Griffing (Cereal Chem., 1925, 2, 325—344).—Flours of various origins were overground either in a laboratory ball mill with flints or on the smooth rolls of an experimental mill, the whole of the flour being reduced to a given fineness. The cold water extract of flour increased progressively with the overgrinding and the percentage of ash and nitrogen in the extracts decreased, so that increase in extract was due mainly to substances that after acid hydrolysis reduce alkaline copper solution, presumably starch. In studying the increased diastatic activity of overground flours it was found that loss of carbon dioxide early in the fermentation was more rapid than for normal flours. If overgrinding was carried to extremes the water absorbed per g. of gluten protein was reduced and the quality of the gluten apparently adversely affected. The glutens washed out from overground flours tended to be freer from non-protein matter, and their rate of swelling in 0·04 N-acetic acid was lessened. From the most severely overground flours no gluten at all could be washed out. Although severely overground flour had increased absorptive powers, mainly due to swelling of starch granules, the baking strength was very distinctly injured, and practically no rising of the dough occurred after "punching," some weak flours even being found incapable of doughing. The exact effect of moderate overgrinding upon absorption has not yet been fully worked out, but it may prove possible to increase absorption without adversely affecting baking strength. D. G. Hewer.

Determination of moisture in wheat and flour. IV. H. Snyder and B. Sullivan (Ind. Eng. Chem., 1926, 18, 272—275; cf. B., 1925, 374).—The flours were dried in an evenly flowing stream of very carefully dried hydrogen, and 58 tests showed an average of 0·54% more moisture by this method than was obtained with the vacuum oven at 100° and 600 mm. or more pressure. Nitrogen and preheated air gave results very slightly lower than hydrogen. The degree of dryness of the medium in which the flours are dried is always of the greatest importance, and any method thus far developed for flour and wheat is a relative rather than absolute expression of moisture content. D. G. Hewer.

Phytin content of foodstuffs. H. P. Averill and C. G. King (J. Amer. Chem. Soc., 1926, 48, 724—728).—The phytin contents of 57 samples of foodstuffs, determined by extracting the finely ground air-dried material (8 g.) with 2% aqueous hydrochloric acid (200 c.c.) for 3 hr. (cf. Rather, A., 1918, ii, 88) and titrating the filtered extract, diluted to a hydrochloric acid concentration of 0·6%, with ferric chloride solution (0·00195 g. of iron per c.c.), using

ammonium thiocyanate as indicator (cf. Heubner and Stadler, A., 1914, ii, 690), are listed. In the case of soya beans and nuts an extraction with ether was carried out prior to the acid extraction, in order to remove oils, the presence of which renders determination of the end-point difficult owing to turbidity. The phytin content of different samples of nuts shows fair uniformity, but that of grains and flours shows considerable variation. Hard and winter wheat flours contain generally more phytin than soft and spring wheat flours, whereas very little difference was observed between wheat and rye in this respect, both for the whole grain and for the flour. The course of purification of phytin by Anderson's method (A., 1920, i, 801) was followed by the above titration procedure; the final product had a phytin content of 99·00%. The phytin content of wheat is lowered appreciably by heating at 105° for 2 hrs., by steaming for ½ hr., by treating with excess of water for 5 hrs., or by moistening for 10 hrs. F. G. WILLSON.

New yeast preparation. E. STERN and H. BECKER (Chem.-Ztg., 1926, **50**, 185—186; cf. U.S.P. 1,574,776, p. 381).—Carefully purified and pressed yeast is plasmolysed with Soxhlet's nutritive sugar at room temperature until the mass is completely liquefied, whereby the whole of the nutritive matter in the yeast cells is converted into an assimilable form; the water content of the yeast is sufficient for plasmolysis without adding further water. The product is heated at 45—50° for 1 hr. to destroy the zymase and subsequently evaporated to dryness at a temperature below 60° and a pressure of 12 mm. The dry residue ("Katazyman") contains 66% of the nutritive sugar carbohydrates and 33% of yeast substance and is rich in lipoids, vitamins, and enzyme carriers. A. R. POWELL.

Vitamins in canned foods. V. Peaches. S. F. KOHMAN, W. H. EDDY, V. CARLSSON, and N. HALLIDAY (Ind. Eng. Chem., 1926, **18**, 302—303; cf. B., 1925, 81).—Experiments with guinea pigs showed that the minimum antiscorbutic dose of raw and canned peaches was about 5 g. per day, but somewhat more gives better growth. The removal of oxygen from the peaches previous to canning gave only an insignificant protection to vitamin-C. Cooking in a covered kettle diminished the vitamin-C content to an estimated proportion of one-quarter to one-fifth. Canned peaches appear to be about one-third as potent in vitamin-A as butter fat. The vitamin-B content was too low to determine owing to the upset of general nutrition by the large quantities required. D. G. HEWER.

Inactivation of vitamin-A by rancid fat. W. C. POWICK (J. Agric. Res., 1926, **31**, 1017—1026). —Feeding experiments with rats are described in which rations containing a large proportion of very rancid lard were used. While the rancid lard did not show actually toxic properties, experiments indicated that its inferiority to fresh lard as a foodstuff was due to its ability to destroy vitamin-A in admixed foods. This destruction of vitamin occurred over a period of several days and is ascribed to oxidation by organic peroxides in the rancid lard. A. G. POLLARD.

Preserved peas in relation to their diameter. E. LASAUSSE (Ann. Falsif., 1926, **19**, 28—40).—The ratio of cellulose and nitrogen to dry extract diminishes as the peas mature, and that of soluble hydrolysable substances to dry extract increases, while that of hydrolysable insoluble substances to dry extract decreases. Thus old dry peas may be distinguished from young green ones (cf. Muttelet, B., 1926, 296). In some cases preserved peas of the same average diameter as green peas contain different proportions of starch and cellulose and absorb different amounts of water on boiling, but peas passing through a sieve of one size may have analytical values very similar to those passing a different size. The district of origin has an effect on the data. Chemical methods of analysis, including those of Muttelet, can only afford an approximate idea of the original sizes of the peas.
D. G. HEWER.

Rapid determination of copper in preserved vegetable products. R. BIAZZO (Annali Chim. Appl., 1926, **16**, 96—98).—The method is based on Spacu's colour reaction (cf. B., 1925, 831). 5 g. of the food are ashed, the residue is treated with a few c.c. of 50% hydrochloric acid, evaporated, and made up to 20 c.c. with water in a separator. Sodium hydroxide solution is added till just alkaline, then the solution is made just acid with acetic acid, and treated with a few drops of concentrated potassium thiocyanate and pyridine solutions. After extraction with 5 c.c. of chloroform, the green colour of the chloroform layer is compared with that obtained from a standard food containing the maximum permissible amount of copper salt.
S. B. TALLANTYRE.

Black currant juice and the reactions of orchil. M. FRANÇOIS and L. SEGUIN (J. Pharm. Chim., 1926, **8**, 241—248).—Natural black currant juice contains about 4% of citric acid and a colouring matter similar to that of red wine. A product sold as "extract of black currant syrup" was found to contain no citric acid, and some 15% of tartaric acid, and was coloured by orchil.
B. W. ANDERSON.

Influence of gases on the conservation of fodder. K. SCHMIDT (Leopoldina, 1926, **1**, 69—72). —The gas drawn from a silo consisted of carbon dioxide, nitrogen, hydrogen, and traces of oxygen and methane. When lucerne was kept in an atmosphere of carbon dioxide below 6° the fermentative production of gas, acetic and butyric acids, and ammonia was much less than in an atmosphere of oxygen or nitrogen. This result was confirmed on a silo. R. K. CANNAN.

Revised net energy values of feeding stuffs for cattle. E. B. FORBES and M. KRISS (J. Agric. Res., 1926, **31**, 1083—1099).—The net energy values of a number of feeding stuffs are corrected

in accordance with the most recent developments of the work of Armsby (1903—1918).
A. G. Pollard.

Variations in absorptive power. Effront.—See XVIII.

Patents.

Milk product. D. D. Peebles (U.S.P. 1,574,165, 23.2.26. Appl., 24.12.23).—Non-fatty solids are removed from milk until the fats and non-fats are in approximately equal proportions and the product (pasteurised or not) is condensed to a content of 20% of water.
D. G. Hewer.

Manufacture of powdered milk. N. H. Christensen (U.S.P. 1,574,233, 23.2.26. Appl., 23.4.25).—Sodium oxalate is added to milk to precipitate a proportion of the calcium, which is then removed. Sodium citrate and an alkali are subsequently added, and the milk is desiccated by passing it in a thin film over a heated roller.
D. G. Hewer.

Casein solids and process of making them. A. A. Dunham, Assr. to Casein Manuf. Co. (U.S.P. 1,575,155, 2.3.26. Appl., 14.2.23).—Casein which has been precipitated by acids, and which contains more than 0·75% of free acid, is treated with substances capable of neutalising acids, in quantity sufficient to reduce the acidity to within the limits of 0·75 and 0·25%.
L. A. Coles.

Fermenting or curing cocoa [cacao] beans. W. B. McLaughlin (U.S.P. 1,575,372, 2.3.26. Appl., 28.12.23).—Cacao beans are maintained out of contact with the air, at a temperature above that at which organised ferments propagate, and below that at which the enzyme of the bean is injured, until the purplish-red colour of the bean changes to a reddish-brown shade, which on drying turns cinnamon brown.
L. A. Coles.

Preservation of foods, beverages, feeding stuffs and the like. T. Sabalitschka (F.P. 596,843, 16.4.25. Conv., 17.4.24).—The esters of carbocyclic acids, *e.g.*, the esters of *p*-hydroxybenzoic acid, are used as preservatives.
H. M. Langton.

Production of bread [containing silica]. G. Senftner (G.P. 423,225, 16.11.24).—Soluble silica is added to the dough used for making bread for patients suffering from arterial sclerosis. The comparatively slow digestion following on the use of such bread promotes intimate contact with every digestive organ and complete resorption of silica results.
H. M. Langton.

Preparation of feeding stuffs. A. Vasseux (Swiss P. 95,512, 4.7.21).—Cellulose-containing substances, particularly such waste materials as brewers' grains, malt combs, exhausted beet slices, are treated with acids, such as hydrochloric or sulphuric, or acid salts, such as bisulphites or bisulphates, whereby carbohydrates contained therein are converted into water-soluble assimilable products.
H. M. Langton.

Stabilising the iron in mineral waters. A. E. Gerard (F.P. 598,364, 19.5.25. Conv., 10.7.24).—The iron generally exists in solution as ferrous bicarbonate which gives rise to deposits in the bottle. This is prevented by adding a small quantity of an organic acid, citric, tartaric, and acetic acids being mentioned.
E. S. Kreis.

Processes of making butter substitutes. Milk Oil Corp., Assees. of C. E. North (E.P. 232,916, 16.12.24. Conv., 23.4.24).—See U.S.P. 1,530,511; B., 1925, 470.

Storage of vegetable produce. J. McL. Thompson (U.S.P. 1,578,218, 23.3.26. Appl., 5.12.25).—See E.P. 240,507; B., 1925, 968.

Cheese. Emulsification and pasteurisation of cheese. Pasteurising cheese. W. J. Mellersh-Jackson. From Swift & Co. (E.P. 249,238–41, 30.12.24).

Yeast preparation (U.S.P. 1,574,776).—See XVIII.

Theobromine from cacao waste (G.P. 423,761).—See XX.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Application of micro-methods to control work in pharmaceutical manufacturing. W. Figdor (Amer. J. Pharm., 1926, 98, 157—162).—Alkali salts of organic acids are determined by heating 0·04—0·08 g. from above in a covered platinum crucible, after moistening with strong sulphuric acid. The residue is re-moistened and reheated till it is white, ammonium carbonate is added, and the crucible ignited to dull redness for several minutes. The ammonium carbonate treatment is repeated till constant weight is attained. The ash in drugs is determined by igniting 20—40 mg. of the finely ground drug in a Pregl micro-muffle in a small platinum boat for 2 min. Residues in tinctures and fluid extracts are determined by evaporating 2 c.c. of the preparation on the steam bath for 5 min., and then drying at 110° for about 3 hrs. to constant weight. Esters and menthol in oil of peppermint are determined in 2 c.c. of oil by the U.S.P. method, and methyl sulphide may be detected by pouring 0·5 c.c. of the oil on to 2 c.c. of Nessler's reagent, a yellow zone or precipitate being formed. The U.S.P. method is applied to the determination of non-volatile extractives soluble in ether or light petroleum by taking 1 g. of substance and 150 c.c. of solvent, using a special Soxhlet extractor in which the thimble is heated by the vapour of the solvent. The method of Dieterle (Arch. Pharm., 1923, 261, 77) for the determination of alkaloids in drugs and alkaloid preparations is thus modified, *e.g.*, in the case of ipecacuanha root: 0·5 g. of finely ground root is shaken for 10 min. with 10 c.c. of sodium carbonate solution and 15 c.c. of ether. 0·3—0·4 g. of tragacanth is added and the liquid exhaustively extracted with ether. The extract is evaporated, treated with

5 c.c. of 0·1 N-sulphuric acid, and titrated with 0·1 N-potassium hydroxide, using methyl-red as indicator.
B. FULLMAN.

Separation of the cinchona alkaloids. J. MESSNER (Pharm. Zentr., 1926, **67**, 146—147).—A theoretical discussion of the author's method of distinguishing the cinchona alkaloids (B., 1903, 712), which has recently been made the basis of a method for the quantitative separation of quinine from other cinchona alkaloids (David, B., 1926, 172). On treatment with monosodium phosphate quinine salts give a precipitate of quinine phosphate. The precipitates obtained from solutions of salts of the cinchona alkaloids on treatment with disodium hydrogen phosphate consist presumably of the free bases, since they are soluble in ether.
B. FULLMAN.

Evaluation of podophyllin. R. EDER and W. SCHNEITER (Pharm. Acta Helv., 1926, **1**, 15—24; Chem. Zentr., 1926, I., 2027).—Podophyllin (about 0·5 g.) is shaken repeatedly with 15 c.c. of chloroform during ½ hr., 10 c.c. of the filtered solution are poured into 50 g. of light petroleum contained in a weighed Erlenmeyer flask, and the precipitate is collected on an air-dried, tared, flat filter paper of 8 cm., diam., and washed with 20 c.c. of light petroleum. After drying for 1 hr. at about 70° and keeping for 1 hr. in the air, the flask and filter are weighed. The proportion of residue, the weighed amount corresponding to two-thirds of the podophyllin taken, should not be less than 40%. A glass filter funnel may be used instead of the filter paper, this being dried at 70° as before, and cooled in a desiccator. Alternatively, the precipitate is returned to the flask, the last traces being washed in with 10 c.c. of acetone; the acetone is then distilled off, the residue dried by blowing dry air through the flask for ½ hr. while it is heated on a water-bath, and the flask cooled in a desiccator before weighing.
L. A. COLES.

Evaluation of commercial vanilla oleoresins. J. B. WILSON and J. W. SALE (Ind. Eng. Chem., 1926, **18**, 283—285).—Authentic vanilla oleoresins for use as standards were prepared by extraction of Mexican, Bourbon, and Tahiti beans with 47·5% ethyl alcohol, and concentrating to d^{25} 1·3. Other samples were prepared from Bourbon beans by extraction with 65% alcohol, and 65% alcohol plus 12% glycerol, and the partially extracted beans were then exhausted with 47·5% alcohol to furnish yet another sample. A portion of the standard Bourbon oleoresin was also mixed with caramel, vanillin, coumarin, and glycerol, and the other samples used for analysis were commercial oleoresins. Analytical data are tabulated for all these samples. The following determinations are recommended—on the well-mixed oleoresin, ash and total alkalinity of ash; on a 95% alcohol extract, non-volatile solids at 100°, vanillin (caramel interferes with the Folin and Denis method on 47·5% alcoholic extract), glycerol; on the water extract of the oleoresin after 95% alcohol extraction, non-volatile solids at 100°, and caramel; and on a 47·5% alcoholic extract, vanilla resin and the Wichmann lead number.
D. G. HEWER.

Odour and chemical constitution in the benzthiazoles. M. T. BOGERT and A. STULL (Amer. Perfumer, 1925, **20**, 453; Chem. Zentr., 1926, **97**, 1., 668; cf. B., 1922, 664 A; A., 1926, 183, 310).—The 2-p-tolyl, α-furyl, and α-tolyl derivatives of benzthiazole have a similar odour (resembling that of tea-roses) to 2-phenylbenzthiazole (Hofmann, A., 1880, 386). The vanillin and piperonal derivatives have a weaker odour and p-phenylenebisbenzthiazole is practically odourless.
E. H. SHARPLES.

Instability of the sodium salt of benzoyl hydroperoxide. A. J. GELARIE and F. R. GREENBAUM (Amer. J. Pharm., 1926, **98**, 163—166).—An aqueous solution of pure benzoyl hydroperoxide decomposes to the extent of 95% in 24 hrs., decomposition being complete in 48 hrs. Carbamide, copper sulphate, hexamethylenetetramine, sodium cinnamate, and the sodium soaps of castor oil, cod liver oil, olive oil, and chaulmoogra oil accelerate the decomposition.
B. FULLMAN.

Acidimetric determination of both components in *Hydrargyrum oxycyanatum*. E. RUPP (Pharm. Zentr., 1926, **67**, 145—146).—Mercury oxycyanide may be thus determined: 0·3 g. of oxycyanide and 0·5 g. of sodium chloride are dissolved in 40—50 c.c. of lukewarm water and the solution is cooled, and titrated with 0·1 N-hydrochloric acid in the presence of 2 or 3 drops of methyl-orange till the first change from yellow to orange occurs. This gives the oxide component. On addition of 1·5—2 g. of sodium thiosulphate the mercury cyanide reacts giving sodium cyanide, which is titrated with 0·1 N-hydrochloric acid to a pink end-point. Commercial preparations do not give equal molecular quantities of both components, the oxide being in defect and the cyanide in excess. The second titration is only necessary when foreign substances such as sodium chloride are present.
B. FULLMAN.

Use of glycogen in the preparation of colloidal metals. L. HUGOUNENQ and J. LOISELEUR (Compt. rend., 1926, **182**, 851—852).—Colloidal solutions of the oxides of many metals (*e.g.*, manganese, copper, iron) are readily prepared by adding the nitrate of the metal to an alkaline glycogen solution. In the cases of gold, platinum, palladium, mercury, silver, and rhodium, the solutions on boiling yield colloidal solutions of the metal. Colloidal solutions of bismuth in gum arabic have no advantage over other bismuth preparations in the treatment of syphilis.
S. K. TWEEDY.

Production of pure absolute alcohol. R. E. LYONS and L. T. SMITH (Science, 1925, **62**, 224—225).—To 100 g. of the alcohol (95·02 vol.-%) are added 17·5 g. of granular (20-mesh) calcium carbide (81·1%) and the mixture is boiled on a water-bath for 30 min. under a reflux condenser, 0·5—1 g. of anhydrous copper sulphate then being added to remove dissolved

acetylene and sulphur compounds. After boiling for a further 15 min. the alcohol is removed by distillation. Alcohol (99·86 vol.-%) in 98·66% of the theoretical yield, free from acetylene, sulphur compounds, aldehyde, and free acid, was obtained in an example. A. A. Eldridge.

Arsenobenzenes, their composition and toxicity, the nature of the substitution, the value of the DM index. F. De Myttenaere (Boll. chim. farm., 1925, 64, 641—649).—The work of Valeur and Launoy (J. Pharm. Chim., 1924, 366; B., 1925, 113) and of Patta (B., 1925, 826) is criticised, and suggestions are made concerning the particulars which should be stated on the labels of pharmaceutical preparations of arsenobenzenes.
T. H. Pope.

See also A., April, 350, **Colloid chemistry of bismuth** (Gutbier). 351, **Preparation of colloidal silver by electrolysis** (Lottermoser and Bausch). 375, **Determination of nitrogen in organic compounds by Kjeldahl's method** (Andersen and Jensen). 381, **Methyl and ethyl iodides from the corresponding toluenesulphonates** (Peacock and Menon). 394, **2:4-Dinitrophenylhydrazine as a reagent for aldehydes and ketones** (Brady and Elsmie). 404, **Syntheses of o-vanillin and o-veratraldehyde** (Mauthner). 405, **Purification of xylene for use as solvent for catalytic reduction** (Zetzsche and Arnd); **Utility of various solvents for catalytic synthesis of aldehydes from acid chlorides** (Zetzsche and others).

Patents.

Manufacture of oxalic acid from wood. R. Falck (G.P. 419,911, 20.9.22; cf. Falck and van Beyma thoe Kingma, B., 1924, 649).—Oxalic acid is produced by subjecting mixtures of wood with calcium carbonate, or with calcium oxide and cheap vegetable material, to the action of wood-destroying fungi. For example, mixtures of spruce shavings or sawdust with calcium carbonate, which may also contain chopped straw or may be worked up with dextrin and water into briquettes, are decomposed by the action at 15° of the fungus, *Coniophora cerebella*. After one month, the calcium carbonate is converted almost entirely into calcium oxalate.
L. A. Coles.

Preparation of acetylsalicylic acid. Deutsche Gasglühlicht-Auer-Ges., Assees. of S. Merzbacher (G.P. 421,021, 23.6.20).—The compound is prepared by heating salicylic acid with ethylidene diacetate, without the addition of catalysts. For example, a solution of salicylic acid in ethylidene diacetate is heated for 5 hrs. under a reflux condenser maintained at such a temperature that ethylidene diacetate and acetic acid condense and flow back into the solution, whilst acetaldehyde escapes as vapour. The product, after removal of acetic acid and a portion of the excess ethylidene diacetate by distillation, sets to a crystalline mass of acetylsalicylic acid, which is purified by draining and crystallisation, a yield of about 70% being obtained. The acetaldehyde can be oxidised to acetic acid, which is then worked up to ethylidene diacetate by treatment with acetylene.
L. A. Coles.

Pharmaceutical compound. Farbenfabr. vorm F. Bayer und Co., Assees. of H. Hahl (U.S.P. 1,561,535, 17.11.25. Appl., 27.6.25.).—A solution of thiosalicylic acid is neutralised with sodium carbonate and saturated with freshly precipitated antimony oxide, and the solution evaporated to dryness. The resulting antimonysodium salt $Sb(S{\cdot}C_6H_4{\cdot}CO_2Na)_3$, is of value as a spirillocide. An analogous compound is obtainable from thioglycollic acid.
T. S. Wheeler.

Preparation of 2-phenyl-4-aminoethylquinoline. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of M. Dohrn and C. Zöllner (G.P. 421,087, 19.11.22).—2-Phenyl-4-methylquinoline is condensed with chloral, the product is hydrolysed, yielding a substituted acrylic acid, this is converted by treatment with reducing agents into the corresponding propionic acid derivative, which is converted into 2-phenyl-4-aminoethylquinoline, *e.g.*, after intermediate conversion into the hydrazide or acid amide. For example, on heating 2-phenyl-4-methylquinoline (flavoline) for several hours at 100° with chloral in the presence of xylene, the methyl group is converted into the $-CH_2.CH(OH).CCl_3$ group, yielding 2-phenyl-4-γ-trichloro-β-hydroxypropylquinoline, m.p. 202°, which on hydrolysis with 20% alcoholic potassium hydroxide and subsequent acidification yields 2-phenylquinolyl-4-acrylic acid, m.p. 205—206°, and this is converted into 2-phenylquinolyl-4-propionic acid, m.p. 215—216°, by heating with acetic acid, concentrated hydriodic acid, and red phosphorus. The propionic acid derivative is converted successively into the ethyl ester; the hydrazide, m.p. 158—159°, by heating with hydrazine and absolute alcohol; the azide, by treatment with nitrous acid; the urethane, m.p. 89—90°, by heating with ether and absolute alcohol; and into 2-phenyl-4-aminoethylquinoline hydrochloride (decomp. at 235°), by heating for several hours with concentrated hydrochloric acid. Alternatively, the propionic acid derivative is converted successively into the acid chloride, by treatment with sulphuryl chloride; the amide, m.p. 197°, by treatment with 25% ammonia; and the amine by the action of bromine and aqueous potassium hydroxide. The 2-phenyl-4-aminoethylquinoline is of therapeutic value.
L. A. Coles.

Preparation of glycerophosphates. J. D. Riedel A.-G., Assees. of R. Rosenbusch (G.P. 421,216, 20.12.22).—Material extracted from soya-bean press cake by means of organic solvents, is treated, preferably after removal of the solvent and before or after removal of lecithin and oil, with alkalis or alkaline-earths. After neutralisation and separation of fatty acid salts, and neutralisation of excess basic material, which is preferably converted into insoluble salts, glycerophosphates are recovered from the residual solution by the usual method. The product is chemically pure and optically active.

For example, the product obtained by extracting the cake with alcohol and removing the solvent is stirred for several hours at 50—70° with saturated barium hydroxide solution. After filtration, excess barium hydroxide in the solution is precipitated by passing in carbon dioxide, the barium carbonate is removed, the solution is concentrated, and the barium glycerophosphate is converted by the addition of calcium sulphate into the corresponding calcium salt, which separates as white crystals on boiling the solution. L. A. COLES.

Preparation of codeinone. E. MERCK, CHEM. FABR., Assees. of W. KRAUSS (G.P. 421,217, 19.10.23; Swiss P. 111,996, 30.7.24).—The compound is prepared by treating methylcodeine, obtained as described in G.P. 418,391 (cf. B., 1926, 216), with chromium trioxide in the presence of weak acids, *e.g.*, acetic acid. For example, aqueous chromium trioxide solution is added to a solution of methylcodeine in 20% acetic acid, whereby the temperature rises to 40—50°. On cooling strongly, codeinone chromate crystallises out, and is decomposed by treatment with ammonia, codeinone being obtained by extraction with ether and evaporation of the solvent. L. A. COLES.

Preparation of phenylsulphur chloride [chlorothiolbenzene] and its homologues. H. LECHER and F. HOLSCHNEIDER (G.P. 423,232, 23.3.24; cf. A., 1924, i, 728).—The compounds are prepared by the action of chlorine upon the corresponding arylmercaptans or diaryl disulphides, preferably in the presence of inert solvents. L. A. COLES.

Preparation of theobromine from cacao waste. E. TIEDE (G.P. 423,761, 31.5.24).—Cacao waste, after extraction of fat, is heated for about 3 hrs. at 200—300° with stirring, preferably with the addition of heat-conducting material such as finely-divided copper, and is then extracted, *e.g.*, with acetylene tetrachloride, theobromine being precipitated from the extract by the addition of ether. L. A. COLES.

Preparation of a stable product containing acetylsalicylic acid. NYEGARD & Co. A./S. (Nor. P. 39,863, 14.12.23).—The product consists of an intimate mixture of finely-powdered acetylsalicylic acid with a quantity of lithium carbonate at least sufficient to neutralise acetic acid liberated by hydrolysis. L. A. COLES.

Manufacture of methylals. H. WADE. From S. KARPEN & BROS. (E.P. 249,039, 23.10.25).—See U.S.P. 1,566,819; B., 1926, 217.

Manufacturing acetic anhydride. CONSORTIUM FÜR ELEKTROCHEM. IND. G.M.B.H., Assees. of R. MEINGAST and M. MUGDAN (U.S.P. 1,570,514, 19.1.26. Appl., 6.3.23).—See E.P. 194,719 and G.P. 410,363; B., 1924, 34; 1925, 1013.

Production of alcohols from halogenated aldehydes. FARBENFABR. VORM. F. BAYER U. Co., Assees. of H. MEERWEIN (U.S.P. 1,572,742, 9.2.26. Appl., 28.5.25).—See E.P. 235,584; B., 1925, 738.

Recovering oxalate from tree barks. W. A. FRAYMOUTH, Assr. to BHOPAL PRODUCE TRUST, LTD. (U.S.P. 1,577,573, 23.3.26. Appl., 30.11.23).—See E.P. 208,684; B., 1924, 577.

Ethylating amines (U.S.P. 1,570,203).—See IV.

Bread containing silica (G.P. 423,225).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Nature of optical sensitisation and desensitisation. G. KÖGEL and A. STEIGMANN (Z. wiss. Phot., 1926, **24**, 18—31).—The view that sensitising dyes function by acting as halogen acceptors is not in accord with the fact that Eosin, Flavinduline, Methylene Blue, etc., are reduced on exposure to light and that this bleaching is accelerated by "sensitisers" which may be powerful reducing agents with at least one labile hydrogen atom. Bleaching is due to hydration (reduction) of the dye. Methylene Blue–gelatin is very sensitive to light, giving an invisible latent impression which can be developed by bathing in silver nitrate followed by physical development. Exposure of Eosin, Erythrosin, Rhodamine B, Pinachrome, Pinaflavol, and Phenosafranine gives nothing which will reduce silver nitrate, but these dyes are probably more sensitive to long wave-lengths than is ripened silver bromide, which they can affect by a primary reaction. With chlorobromide papers and fast iodobromide plates bathed in Methylene Blue, on exposure to yellow or red light, only the dye and not the silver halide was affected; the latent image was on the dye alone. The bathed fast plate was much more sensitive than the paper, a fact which is due to ripened silver bromide reacting more readily to traces of nuclei (formed from the exposed dye and silver nitrate) than does the unripened salt. It is confirmed that silver bromide accelerates the bleaching of Methylene Blue in light, but only in short wave-lengths. Silver bromide in short wave-length light can activate hydrogen, thereby being reduced and also reducing the dye which takes up part of the activated hydrogen. In long-wave-length light, the dye activates the hydrogen of the sensitiser (*e.g.*, gelatin) and is thereby bleached, independently of silver bromide. Panchromatic sensitisation is explained as follows: Under the influence of light of long wave-length the labile hydrogen of the sensitiser (gelatin) is photochemically activated by the dye which absorbs these wave-lengths, and the activated hydrogen is taken up partly by the silver bromide and partly by the dye adsorbed to it, both being reduced. In short wave-lengths the silver bromide activates the hydrogen. Desensitisers are easily reduced and probably take up hydrogen more readily than sensitisers. Desensitisation is ascribed to the fact that the hydrogen (from the gelatin) activated by the silver bromide is all taken up by the dye, so that reduction of the silver halide cannot occur. Leuco-bases of desensitisers cannot desensitise. Free silver has only a slight effect in accelerating the

photochemical oxidation of metol, as compared with the effect of silver bromide. Silver bromide is thus inferior to silver in its power of activating hydrogen in short-wave-length light. It is suggested that ripening reduction nuclei and exposure nuclei can sensitise silver bromide if there are chemical forces between the silver of the nuclei and the silver halide which loosen the silver bromide molecule, giving weak points where light or the activated hydrogen can act readily. Gelatin owes its strong sensitising power to the fact that it contains hydrogen capable of activation. W. Clark.

Determination of silver in photographic emulsions. (A) A. Steigmann, (B) W. Meidinger (Z. wiss. Phot., 1926, 24, 15—16, 16—17).—(A) The author was the first to recommend the use of Liebig's cyanometric method for the determination of silver in photographic emulsions (cf. Eggert, B., 1924, 235; Meidinger, B., 1925, 23, 282). (B) Reply to Steigmann, quoting historical data. W. Clark.

Influence of strong heating on the properties of photographic dry plates. H. M. Kellner (Z. wiss. Phot., 1926, 24, 63—78).—In general, heating a plate results in an increased density of the developed negative. Heating during exposure has an especially strong action; heating before exposure gives a marked speed increase with plates of low sensitivity; heating after exposure gives roughly the same effect with all plates, but less marked than heating before exposure. The most pronounced effect is obtained with the emulsion side in contact with the heating surface, except in the case of thinly coated plates, with which the reverse holds. In most cases rise of temperature gives an increase in speed, up to about 90°, above which speed falls again and fogging occurs. Some plates show no such maximum. There seems to be an optimum on increased heating at constant temperature. The effect of heating is decreased if plates are previously dried over phosphorus pentoxide. Under some conditions heating produces a change in spectral sensitivity. In general the position of the sensitivity maximum, and the sensitivity range, are unaltered, but the shape of the curve may be changed. No appreciable alteration in size of the silver bromide grains occurs on heating. The thickness of the emulsion layer appears to increase on heating, during the time that sensitivity is increasing. The number of developed silver grains per unit area is increased by heating, especially for low light intensities. Heating results in a closer packing of the developed silver grains in the surface layers of the emulsion. A theory of ripening is proposed, according to which the valency electron of a bromine ion in the silver bromide lattice is normally present in the innermost orbit, corresponding with the lowest energy content. Exposure shifts the electron from this orbit to one of a higher energy state, and sufficient light energy can cause complete dissociation, the electron going to a silver ion as acceptor, and forming a latent image nucleus. Heating can bring about the same effect, so that ripening consists in the formation of activated bromine ions; if ripening is too prolonged, the bromine ions lose their electrons to silver ions and fogging occurs. Heating could possibly also dissociate a bromine ion activated by exposure. W. Clark.

Topographical relations in development. Lüppo-Cramer (Z. wiss. Phot., 1926, 24, 1—12).—Grain surface development, grain depth development, and film depth development are considered. The explanation that slow-working developers are suitable for strong over-exposures owing to their having time to diffuse into the depth of the film before the surface layer has been appreciably reduced, is not entirely correct, because slowing development by adding bicarbonate, dilution, etc., instead of using bromide, does not give correct development of over-exposures. With solarised and over-exposed images there is no difference in the appearance of the image whether exposure is made from the glass or from the film side. Local distribution of image in over-exposures does not play such a big part as to explain the difference in action of rapid and slow developers. The image on physically developed silver iodide–gelatin plates is on the surface, but with silver bromide–gelatin this is not so. This difference is explained as due to the latent image on silver iodide consisting of smaller, more numerous and more active amicrons than on silver bromide. With a certain fine-grain diapositive silver bromide emulsion, solarisation was obtained on physically developing after fixing. That this is not observed with coarse-grained emulsions is supposed to be due to nuclei in the grain interiors which are set free on fixing, while with small grains the nuclei are all on the surface. Solarisation depends less on grain size than on the inner structure of the grain and the kind of development. W. Clark.

Development paradoxes. Lüppo-Cramer (Z. wiss. Phot., 1926, 24, 13—15).—A 0·05% solution of pure diaminophenol hydrochloride (amidol) develops much more rapidly than 0·5%, 1%, and 2% solutions. With 1% solution the image appears first on the back side of the film, while the dilute solution develops normally from the front side. The "depth development" is explained as due to the gelatin absorbing the hydrochloric acid of the developer so that in the depth of the film the developer is relatively less acid and so more active. Diluted amidol develops more quickly than the concentrated with collodion as well as gelatin plates, and the hydrochlorides of triaminobenzene, triaminotoluene, and diaminoresorcinol, and metol act like amidol. With 1 : 100,000 amidol, which normally develops a good image, addition of sulphite or sulphite–carbonate retards development. Triaminobenzene and triaminotoluene show the same effects, but although these developers with no additions develop powerfully, in presence of bromide they destroy the latent image. Concentrated (1%) amidol containing 1% of potassium bromide has a more powerful destructive action than the same developer diluted a hundredfold. Similar results were obtained with triaminobenzene and metol, but to a less degree. 1% ferrous sulphate solution develops fully exposed silver bromide

diapositive plates more quickly and more strongly than a 30% solution. W. Clark.

Theory of the Carbro process. F. J. Tritton (Phot. J., 1926, 66, 126—133).—The reactions occurring in all stages of the single-bath and combined-bath Carbro process are considered in detail. Potassium ferricyanide is not regarded as a catalyst for the reaction between silver and chromic acid. When the bromide and tissue are squeegeed into contact, if an acid bath has been used, the gelatin of the tissue will continue swelling, while that of the bromide will not have much tendency to swell, so that there will be a general direction of diffusion from the bromide print into the tissue. The ferrocyanide formed in the reactions will thus diffuse normally into the tissue, so that sharp images will be obtained. The effects of varying times of treatment in and temperature of the different baths, and the thickness of tissue, and hardness of the bromide, are discussed in relation to the theoretical considerations advanced. W. Clark.

New physical method for the examination of gelatin. T. Thorne Baker and L. F. Davidson (Phot. J., 1926, 66, 120—125).—The ultra-violet absorption of 1% gelatin sols was studied at p_H values of 3·5, 10·0, and 6·1. If the p_H of an ash-free gelatin is made more or less than 4·7, the absorption curve lies for the most part to the long wave-length or short wave-length side respectively of the absorption curve of the ash-free gelatin; *i.e.*, for a given thickness of gelatin, a p_H on the acid side of the isoelectric point gives greater transparency to the ultra-violet. A gelatin extracted by the centrifuge from a silver bromide photographic emulsion gave an absorption curve identical in shape with that of the original gelatin. A simplified photographic method for determining absorption spectra is described. W. Clark.

Patents.

Compensating the copying intensity of kinematograph films. E. Lehmann (U.S.P. 1,568,511, 5.1.26. Appl., 27.6.24).—Kinematograph films are compensated for differences in density by impregnating the gelatin covering of the film with a colouring agent which is incapable of yielding fast colorations with it. S. Binning.

Manufacture of gelatin suitable for [photographic] emulsions. A. Jenny and J. Angerstein, Assrs. to Amer. Electro-Osmosis Corp. (U.S.P. 1,577,642, 23.3.26. Appl., 13.3.24).—See E.P. 210,748; B., 1924, 655.

XXII.—EXPLOSIVES; MATCHES.

Determination of tetryl in explosives. K. Lehmstedt (Z. angew. Chem., 1926, 39, 379—380).—Tetryl mixed with other explosives, *e.g.*, trinitrotoluene, is determined by measuring the volume of nitric oxide evolved in a nitrometer by the reduction of the nitroamino-group to the amino-group by mercury and strong sulphuric acid. This method is more rapid and satisfactory than the reduction by ferrous chloride and hydrochloric acid, which gives high results in the presence of tetranitroaniline. B. W. Clarke.

Deflagration products of smokeless powder. H. C. Knight and D. C. Walton (Ind. Eng. Chem., 1926, 18, 287—291).—Experiments were made at Edgewood Arsenal for the U.S. Navy Department to determine the composition of the gases formed when smokeless powder was burnt in a gun turret where excessive pressure could not develop and complete combustion was prevented by lack of sufficient air. The toxicity of the products and the length of time during which the toxicity lasted were also investigated. Charges of 32 kg. of a single-base powder were ignited in a horizontal cylindrical steel tank, 8 ft. in diam. and 18 ft. long. Samples of the combustion gases were withdrawn for analysis and measurements of the temperature and pressure within the cylinder were recorded. The burning took place in two stages. The first consisted of the combustion of the powder, resulting in a temperature of about 750°, and a pressure of 3·5—4 in. of water in the cylinder; the second comprised the burning of the combustible gases in the cylinder by air drawn in when the cylinder contents cooled. Analyses of gases showed that there was a period when no oxygen was present, and the percentages of carbon monoxide and nitric oxide might be as much as 30% and 10%, respectively. The products of the direct deflagration reached their greatest concentration in 15—20 sec. after ignition, and the secondary combustion began after another 10—15 sec. Toxicity of the gases was due to carbon monoxide and oxides of nitrogen. Animals which escaped carbon monoxide poisoning often developed pulmonary œdema from breathing the nitrous fumes. The only effect of a water spray inside the cylinder was to reduce the temperature, the amount of carbon monoxide and nitrous fumes remaining unaltered. S. Binning.

Patents.

Explosive. T. L. Davis (U.S.P. 1,568,502, 5.1.26. Appl., 8.3.24).—The explosive contains hexanitrocarbanilide as one of its constituents. S. Binning.

Preparation of liquid-air blasting cartridges for use in presence of coal dust and firedamp. Sprengluft Ges.m.b.H. (G.P. 420,218, 17.11.23).—Stoichiometric proportions of two dissociable salts, *e.g.*, ammonium chloride and calcium carbonate, are added to the carbonaceous material. As a result of the detonation ammonium carbonate and also an undissociable salt, calcium chloride, are formed. S. Binning.

XXIII.—SANITATION; WATER PURIFICATION.

Removal of dissolved oxygen from liquids [water]. Detection of small quantities of oxygen in gas mixtures. E. Schulek (Z. anal. Chem., 1926, 68, 22—24).—The liquid to be freed from oxygen is placed in a glass cylinder provided with a stopcock at the top and bottom, and with a

plate of porous sintered glass just above the lower stopcock. By connecting the upper stopcock to the vacuum pump a stream of hydrogen or nitrogen is drawn through the liquid for 3 min. in which time all trace of oxygen is removed owing to the fineness of the gas bubbles produced in the liquid by drawing the gas through the porous plate. In order to detect a trace of oxygen in a mixture of gases an air-free solution of manganous chloride is prepared as above, and after addition of sodium hydroxide and iodide, the gas to be tested is drawn through the solution as slowly as possible. On the addition of hydrochloric acid and starch a blue colour indicates that the gas contained oxygen. The method will detect 0·02 c.c. of oxygen in 100 c.c. of gas. A. R. POWELL.

Preparation and comparative performance of base-exchange water-softening materials. E. B. HIGGINS and J. P. O'CALLAGHAN (Water and Water Eng., 1925, 27, 392—394).—The natural zeolite "permutit B," prepared from glauconite, possesses practically unlimited base-exchange capacity and is free from the tendency to disintegrate which is inseparable from introduction of large quantities of replaceable sodium into artificial zeolites. Its extremely rapid action in both softening and regeneration allows of the production of an enormously increased amount of water of zero hardness from a given quantity of zeolite by regenerating at shorter intervals. Even the most carefully prepared synthetic materials are much less stable, physically and chemically, than natural base-exchange minerals. The former are, without exception, particularly susceptible to the action of dissolved carbon dioxide, which removes the more soluble portion of the molecule, *i.e.*, that portion responsible for base-exchange property. Natural zeolite under similar conditions is not affected, and completely resists water fully saturated at atmospheric pressure with carbon dioxide for several months. The acid-resistance of natural zeolite is important where corrosive water is to be treated, and also where the water is first partially softened by treatment with lime, in which case deposits of calcium carbonate may be removed by washing with dilute hydrochloric acid without injuring the softening material. The base-exchange activity of natural zeolite contaminated with oil and tar can be fully restored by roasting.

R. E. THOMPSON.

Chlorination of the Manila water supply. E. TAYLOR (Water and Water Eng., 1925, 27, 409—412).—Chlorination of the Manila water supply was commenced in 1914, a dosage of about 0·6 p.p.m. being used. The results obtained were not satisfactory and the practice was adopted of maintaining a residual chlorine content, as determined by the *o*-tolidine test, of 0·05—0·1 p.p.m. throughout the distribution system. This method has given excellent results, maintaining a low bacterial count and almost complete absence of *B. coli*. The chlorine-consuming power of the water varied between 0·4 and 1·4 p.p.m., with a contact period of 4 hr. When the water was turbid the chlorine consumption usually increased.

R. E. THOMPSON.

Iodoform taste of chlorinated water. B. ADAMS (Water and Water Eng., 1925, 27, 435—436).—A concentration of 1 pt. of iodoform in 1000 million pts. of water was found to give a decided taste which was indistinguishable from that due to chlorinated phenol. A trace of alcohol or acetone with iodine in an alkaline solution will give the iodoform reaction, and addition of 0·2 p.p.m. of iodine to water containing 1 p.p.m. of alcohol or acetone will give rise to tastes, predominantly iodoform, on keeping. Therefore, if a water contains the necessary organic matter, and iodides from which iodine will be liberated, an iodoform taste will develop on chlorination. Experiments with water derived from deep chalk wells are described in which an iodoform taste resulted, after appreciable lapse of time, from chlorination (0·25 p.p.m.) after addition of 0·05—0·25 p.p.m. of potassium iodide (expressed as iodine), the water apparently normally containing the necessary organic matter for the production of iodoform. As in the case of taste due to phenol, super-chlorination and dechlorination prevented the development of the taste. Potassium permanganate, prior addition of ammonia, and addition of ammonia with the chlorine were ineffective. Further experiments showed that potassium permanganate would reduce and prevent the taste with lower concentrations of iodide. A distinct taste was given by 1 pt. of iodide in 200 million pts. of water. Potassium bromide did not produce an abnormal taste. R. E. THOMPSON.

Chlorine in sewage and waste disposal. J. C. BAKER (Water and Water Eng., 1925, 27, 440—441).—Domestic sewage can usually be treated satisfactorily with 5 to 10 p.p.m. of chlorine to give a reduction of 95 to 99% in bacterial count. The germicidal power of chlorine is not due to direct oxidation, but to the formation of some substance toxic to the organism. Substances of greater oxidising potential than chlorine, *e.g.*, permanganate and peroxide, have much lower germicidal power. The oxidising power of chlorine can be considerably reduced by combination with ammonia to form either nitrogen trichloride or chloramine, without loss of sterilising power and very little alteration in sterilising rate. Chloramine, particularly, has a relatively low oxidising potential. The chloramine probably acts by attacking the lipoid protein surface of the cellular organisms and forming there some substance which interferes with further cell division, thereby stopping regeneration and resulting in death of the organisms. R. E. THOMPSON.

Nitrate determinations [in water and sewage]. G. W. BURKE (J. Amer. Water Works Assoc., 1926, 15, 169—170).—When using the Amer. Publ. Health Assoc. phenoldisulphonic acid standard method for determining nitrates, samples can be accurately matched by one of the standards, when the same amount of phenoldisulphonic acid is added to each of the Nessler tubes containing the standards, as is used to moisten the evaporated residue of the sample. In the reduction method in which the nitrite value (otherwise determined) is subtracted from the nitrate plus nitrite value obtained by

nesslerisation it is necessary to know accurately the value of the nitrite standard solution in terms of ammonia, and this is best determined by reducing a sample of the nitrite standard solution.
W. T. LOCKETT.

Use of chinic [quinic] acid in the differentiation of the colon-aerogenes group. B. H. BUTCHER (J. Amer. Water Works Assoc., 1926, 15, 171—173).—Beijerinck observed that some strains of *B. aerogenes*, under aerobic conditions, produced a red to black coloration in a medium containing quinic acid and a ferric salt, but that no coloration was produced by *B. coli.* The medium used by the author contained 0·5 g. K_2HPO_4, 0·5 g. NH_4Cl or peptone, 0·1 g. $FeCl_3$, and 10·0 g. of calcium quinate per litre of water. It was found preferable to inoculate agar agar slants or plates rather than a liquid medium. At 37° some strains of *B. aerogenes* produced colour within 24 hr.; other strains, which did not give a distinctly positive Voges-Proskauer reaction required 3—4 days. Observations indicated the possibility of using the test to differentiate organisms within the aerogenes group as well as between the coli and aerogenes groups. Of 18 organisms of the aerogenes section, six strains of *B. aerogenes* and five strains of *B. oxytocum* checked with the Voges-Proskauer reaction, whereas six strains of *B. cloacae* and two strains of *B. levans* produced no coloration in the quinate medium. Of twenty-three strains of the coli section tested, none gave any coloration.
W. T. LOCKETT.

Determination of thallium in mouse poisons. F. MACH and W. LEPPER (Z. anal. Chem., 1926, 68, 36—45).—10 g. of the material are heated with nitric and sulphuric acids to destroy organic matter and, after expulsion of the nitric acid and dilution, the thallium is reduced to thallous salt with sulphur dioxide. An excess of ammonia is added to the filtered solution, followed by 25 c.c. of ferric citrate solution and 25 c.c. of magnesia mixture to remove phosphoric acid. The solution is diluted to 400 c.c. and 100 c.c. are filtered off and treated with 25 c.c. of 4% potassium chromate solution. The precipitate is collected next day on a Gooch crucible, washed first with 1% potassium chromate solution, then with 80% acetone, dried for 1 hr. at 120—130° and weighed as Tl_2CrO_4. Precipitation of thallous iodide from acetic acid or ammoniacal solutions is not quite complete. The most accurate method of determining thallium in pure solutions of its salts consists in precipitation of thallic hydroxide by the addition of potassium hydroxide and ferricyanide; the hydroxide is converted into thallic oxide for weighing by heating at 200° to constant weight.
A. R. POWELL.

Determination of dust in the atmosphere. D'ARSONVAL and F. BORDAS (Compt. rend., 1926, 182, 823—825).—Preliminary determinations of the atmospheric dust with a modified Owens' gauge are recorded. The analysis of a dust collected in Paris is given and the detrimental influence of its constituents on the public health is considered.
S. K. TWEEDY.

Arsenic in burner gases and its bearing on Haff disease. GLASER.—See VII.

Utilisation of disinfectants containing mercury. HILGENDORFF.—See XVI.

PATENTS.

Purification of hard water by base-exchanging bodies. E. M. E. FRÉCHOU (E.P. 244,713, 23.9.25. Conv., 19.12.24).—Water is passed through a number of beds of base-exchange material connected in series and provided with means to allow for the methodical circulation of the water through the beds and the regeneration of the material of each of the beds in turn.
W. T. LOCKETT.

[Base-exchange] bodies for the purification of water. E. M. E. FRÉCHOU (E.P. 247,431, 29.4.25).—Base-exchanging substances are prepared by fusion of a mixture of the ashes of mineral fuels, *e.g.*, bituminous or semi-bituminous coals, which yield ashes of relative fusibility, and sodium carbonate. The substances may be readily obtained by projection of a pulverised mixture of coal and sodium carbonate against a non-horizontal wall of a combustion chamber, the wall being previously heated to 1000—1200° so as to obtain combustion of the fuel and fusion of the ash and the soda.
W. T. LOCKETT.

Separating, washing, and discharging comminuted solid material [zeolite] immersed in liquids. C. H. NORDELL (U.S.P. 1,570,854, 26.1.26. Appl., 12.9.22).—An apparatus for washing regenerated zeolite material comprises a chamber fitted at the bottom with a check valve giving admission to a chamber below. Zeolite material mixed with water enters the upper chamber from which it displaces water through an opening in the top. When the pressure in the chamber has risen sufficiently the valve at the bottom opens and the solid falls into the lower chamber from which it displaces water, which passes up through the valve in the opposite direction and washes the solid. The valve then closes and the cycle is repeated.
T. S. WHEELER.

Treatment of household and street waste. K. GERSON (E.P. 247,284, 11.11.24).—Refuse, separated from metallic material and ashes, is fed, without previous drying, to a rapidly running disintegrating device, *e.g.* a beating mill, wherein the fibrous constituents of the garbage are converted into a fluffy fibrous mass (garbage wool), and the non-fibrous constituents are reduced to a powder. The products are then separated. The garbage wool is utilised in the manufacture of pasteboard, paper and the like, or used as a fuel, or is submitted to dry distillation in order to obtain tar and other products. The dust, mixed with water, in which it remains in suspension, is admitted to the sewers.
W. T. LOCKETT.

Insecticides (U.S.P. 1,573,490).—See XVI.

Hydrocyanic acid (G.P. 420,729).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 28, 1926.

I.—GENERAL; PLANT; MACHINERY.

Theory of shaft furnaces. H. Le Chatelier (Compt. rend., 1926, **182**, 735—739).—The theory of heat exchanges in furnaces consisting of a vertical cylinder at the top of which the solid substances to be worked up enter continuously, and hot gases at the bottom, is discussed. Assuming that the heat exchange is not affected by secondary factors and is instantaneous, then if the specific heat of the gases exceeds that of the solids, the latter will leave the furnace at the temperature of the incoming gas. If the reverse is the case, the gases will leave at the temperature of the cold solids. When the specific heats are equal, the behaviour is variable. According as the heat capacity of the solids entering is greater or less than that of the gases entering in the same time, the whole furnace will be at the temperature of the cold solids or of the hot gases.

R. Cuthill.

Solubility of calcium sulphate at boiler-water temperatures. R. E. Hall, J. A. Robb, and C. E. Coleman (J. Amer. Chem. Soc., 1926, **48**, 927—938).—The solubilities in water of anhydrite and soluble anhydrite were determined under conditions obtaining in boilers. The values in g. of $CaSO_4$ per 1000 g. of solution, fell from 0·810 at 100° to 0·065 at 207° in the former case, and from 1·124 at 115° to 0·264 at 185° in the latter. The figures for soluble anhydrite agree with those of Melcher (B., 1910, 150), and of Boyer-Guillon (Ann. Conserv. Arts Métiers, 1900, [iii.], **2**, 189); those for anhydrite agree with Melcher's results at higher temperatures, but do not agree so well at lower temperatures. S. K. Tweedy.

Sodium bicarbonate in dry fire extinguishers. C. Gentsch (Z. angew. Chem., 1926, **39**, 450—451).—Sodium bicarbonate is not instantaneously decomposed at a red heat, as has been stated, for the decomposition requires 3 min. in a crucible heated over a powerful flame, and only reaches 10% in the first 15 sec. The carbon dioxide produced by this decomposition cannot therefore be relied upon for the instantaneous extinguishing of a fire.

B. W. Clarke.

Apparent paradox of plasticity. A. de Waele (Kolloid-Z., 1926, **38**, 257—259).—The method of testing the plasticity of "stand oil," prepared by diluting oils which have been polymerised to the gelation point, in the plastometer may lead to smaller values of ϕ, the volume proportion of the viscous phase, than those obtained by the use of a viscosimeter. It is found that ϕ increases with the radius of the capillary, when the radii are small. An explanation is suggested in terms of the fact that the size of particle of the disperse phase becomes comparable with the radius of the tube. It is therefore necessary to use sufficiently wide tubes for this test.

C. H. D. Clark.

Adsorption from solutions. Sabalitschka and Erdmann.—See XX.

Patents.

Vertical drying and dehydrating apparatus. Baechler, Kiser & Co. (E.P. 241,901, 20.10.25. Conv., 21.10.24).—A vertical cylindrical casing contains a number of inverted truncated conical hoppers, through which the material drops in succession on to flat plates arranged below the small ends of the cones. Between each cone and plate rotates a runner, comprising a number of curved (involute) blades which work the material from the centre to the circumference of the plate, one blade at least being long enough to push the material over the edge of the plate, whence it drops into the next cone. The drying is effected by hot air admitted to the bottom and exhausted at the top of the casing. The rate of treatment can be adjusted by varying the distance between the cones and plates. B. M. Venables.

Centrifugal dryer. A. McKay (U.S.P. 1,576,528, 16.3.26. Appl., 18.10.24).—A number of dished plates are mounted on a vertical rotating shaft inside a casing through which a drying medium is passed, a fixed, inverted conical hopper being provided between each pair of plates. Each plate has a raised centre and periphery, and is flatter at the centre and turned up to a greater height at the periphery than the one above it. A. Geake.

Centrifugal machines. Aktiebolaget Separator (E.P. 244,737, 23.11.25. Conv., 19.12.24).—In a centrifugal purifying or separating device for liquids, at least one constituent of which is volatile, and may form an explosive or noxious mixture with air, means are provided whereby a current of air automatically flows outwards through the outlet for one constituent (preferably the most volatile one) and inwards through the outlets for the other separated constituent and for excess feed. The volatile constituent and air are conducted away together through a pipe leading to a room apart from the separator room, and enter a closed tank above the level of the liquid therein, the liquid being drawn off through an inverted siphon pipe forming a liquid seal, and the noxious air being allowed to exhaust through a high-level outlet, preferably including a condenser which will recover some of the volatile liquid. The current of air is produced either by fan

blades running with the separating bowl, or by having the final outlets from the bowl for the volatile liquid at a greater radial distance from the centre than the outlets for the other constituents. B. M. VENABLES.

Centrifuges. AKTIEBOLAGET SEPARATOR (E.P. 246,831, 22.1.26. Conv., 2.2.25).—A centrifugal separator which is provided with an upper feed vessel and a collector with outlet for excess feed, is also provided with an overflow, adjustable as regards height, from the stationary feed vessel into the collector, so that the untreated liquor or pulp is fed at a head not exceeding a definite value.
B. M. VENABLES.

Centrifugal machines. L. SALCINES (E.P. 248,022, 26.9.24).—The apparatus comprises a perforated outer rotating basket through which the filtered liquor passes and an inner conveying and scraping drum which rotates at a slightly different speed to the basket. The inner drum serves to convey the deposited solid matter to outlets at the bottom of the basket, the bulk of the material being conveyed by a spiral worm on the surface of the drum; the outer edges of the spiral are some distance from the filtering surface of the basket, but scraping projections are pushed outwards to within a short distance of the filtering surface by means of compressed air supplied at intervals to the interior of the drum. After the scrapers have removed as much as possible of the deposited solids the filter surface is finally cleaned by jets of steam or compressed air projected through nozzles also situated on the outer edges of the conveying worm. The rate of feed of material is regulated by pistons in the feed shoot, which control electrical contacts, which in turn control a feed valve operated by a solenoid. B. M. VENABLES.

[Ring and roll] crushers. ACIÉRIES RÉUNIES DE BURBACH-EICH-DUDELANGE SOC. ANON. (E.P. 244,073, 17.11.25. Conv., 8.12.24).—In a crushing machine of the ring and roller type the shaft of the top roller is supported by fixed bearings on one side only of the crushing zone and is driven by the source of power. The ring is driven by the top roll only, and the other two rolls are driven by the ring. The bearings for the two lower rolls are mounted entirely on springs, a system of three springs being used near the rollers to produce the grinding pressure, and five springs at the outer ends of the roll shafts, so arranged that the bearings can give in any direction, radial or axial, but the rolls are always pressed outwards against the ring. B. M. VENABLES.

Ball or tube mills for grinding or crushing. F. W. DAVIS (E.P. 249,045, 27.11.25).—For drying materials undergoing the process of grinding, a ball mill is provided with air-inlet holes in the shell, near the end at which the material is admitted. Such holes may be guarded on the outside by hoods facing in the direction of rotation and on the inside by baffles sloping in the direction of rotation and interlocking with each other to form steps, with spaces at each step for the inlet of air. The air is exhausted with the ground material and if desired may be heated before entering. B. M. VENABLES.

Recovery of the solid constituents of liquids containing them. H. N. MCLEOD (E.P. 248,081, 28.11.24).—The liquid to be treated is pumped first through one tube of a three-tube regenerative system and is then heated above its boiling point (*e.g.*, by passing it through the jacket of an internal-combustion engine which works the pump) and sprayed into a chamber which may be heated by passing the exhaust gases of the engine through a jacket around it. The dry powder is removed from the spray chamber by mechanical means and the vapour from the chamber and the exhaust gases from the jacket are passed through the other two tubes respectively of the regenerator, the vapour being thus condensed to a liquid, which has been rendered innocuous by the use of a sufficient degree of heat or by the addition of a chemical agent, or both. The process is particularly applicable to waste products from butter and cheese factories. B. M. VENABLES.

Crystalliser. M. J. KERMER (U.S.P. 1,575,584, 2.3.26. Appl., 2.4.24).—A heating or cooling agent passes through the outer of two concentric chambers or conduits and the material to be crystallised is conveyed through the inner one by means, such as a double screw conveyor, which moves the material in both directions, but to a greater extent in one direction. Both sets of conveyor blades are mounted on the same shaft, which is supported at intervals by bearings inside the inner conduit. B. M. VENABLES.

Dialysing and evaporating membranes [from nitrocellulose]. J. M. LOONEY, Assr. to P. A. KOBER (U.S.P. 1,576,523, 16.3.26. Appl., 23.11.21; cf. B., 1922, 271 A).—Permeable membranes are prepared by mixing nitrocellulose and ethyl alcohol, dissolving the nitrocellulose by agitating the mixture with ether, adding a water-soluble ester, spreading, and removing the solvent. A. GEAKE.

Liquefaction of gases. BADISCHE ANILIN- & SODA-FABR. (F.P. 600,028, 24.6.25. Conv., 20.6.24).—The gases are dried and cooled by treatment with calcium chloride and liquid ammonia, and are then freed from the last traces of moisture by passing through porous non-vegetable matter such as silica gel, permutit, or silicates, prior to introduction into the liquefaction apparatus. E. S. KREIS.

Drying of road-making aggregates and other broken or granular materials. J. F. WAKE (E.P. 249,279, 23.9.24).

See also pages 397, **Securing intimate contact between liquids** (U.S.P. 1,575,116). 400, **Filters** (E.P. 248,044). 405, **Heat interchangers** (E.P. 248,999). 408, **Furnaces** (E.P. 248,036). 409, **Rotary kilns** (E.P. 248,079).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Composition of coal: plant cuticles in coal. V. H. LEGG and R. V. WHEELER (Safety in Mines Res. Bd. Paper No. 17, 1926, 10 pp.).—A comparison has been made of the fossil plant cuticles found in

bituminous coal with cuticles from modern plants (cf. B., 1925, 619). Fossil cuticles can be separated from bituminous coal by mild oxidation followed by extraction with alkali, but, a sufficient supply being more readily obtainable from Russian " paper coal," this has been used as the main source of material. Geological considerations show that the cuticles from both sources are probably closely allied. When freed from ulmin material and from a small quantity of waxy constituents that they contain, the cuticles are attacked only to a slight extent by alkalis, differing in this respect from modern cuticles (*loc. cit.*). The difference is due probably only to the elimination of the external acid groupings of the cuticle rather than to any profound change in structure. On distillation in a vacuum the fossil cuticles yield almost as high a proportion of tar (40%) as do the modern cuticles. The chemical composition of the cuticles differs considerably from that of the ulmic ground mass of the coal in which they are embedded.

A. B. MANNING.

Fundamental study of Japanese coal. III. Relation between microscopic structure and chemical properties. C. IWASAKI (Tech. Rep. Tôhoku, 1926, 6, 1—84; cf. B., 1925, 194).—Soya, Miruto, and Miike coals were examined under the microscope by transmitted and reflected light and by means of *X*-rays. The Soya coal is a black lignite with 20% of moisture; plant tissues such as bark, cork, bast-fibres, resins, stone cells, cuticles, and bark-parenchyma were detected. Miruto coal is a low-grade bituminous coal with only traces of plant tissues. Miike coal is a sub-cannel coal consisting of ' dull ' coal with a homogeneous structure. The conversion of the plant substances to humic matter is traced by photographs of the various coals. More sulphur was found in the ' bright ' than in the ' dull ' coal and the Miike coal was particularly rich in ' sulphate ' sulphur, viz., 0·56%. The ash from the ' bright ' coal in the Soya coal contained 27·99% Fe_2O_3 as compared with 6·32% in the ash from the ' dull ' coal; the silica content was 58·43% in the ash from the ' dull ' coal, and 7·13% in the ash from the ' bright ' coal. Curves showing the volatile matter evolved every 50° from 150° to 1100° gave peaks at 250°, 400°, and 550°. The first and second peaks are due to the woody and plant substances present and the third to the transformed humic matter.

A. C. MONKHOUSE.

Fungi which grow on coal. T. IWASAKI (Tech. Rep. Tôhoku, 1926, 6, 85—94).—The growth of fungi on Yoshioka, Horonubu, Soya, and Machida coals was examined and the following fungi were detected:—*Aspergillus minimus*, *A. oryzæ*, and *Citromyces pfefferianus* (cf. B., 1925, 350). The growth of fungi is less as the geological age of the coal increases, the maximum growth being obtained on the brown lignite. The fungi are richer in growth on the " dull " than on the " bright " coal and for the particular coals examined maximum growth was obtained with *Aspergillus minimus*.

A. C. MONKHOUSE.

Recent investigations on the origin of coal. F. FISCHER (Z. deuts. Geol. Ges., 1925, A., 77, 534—550; Chem. Zentr., 1926, I, 2081).—Wood breaks down under the action of fungi and enzymes into cellulose and lignin. The former is completely decomposed by bacteria and fungi, and disappears, while the latter does not support the growth of these organisms. The lignin only loses its acetyl and methoxyl groups, with formation of humic acids and by loss of water, anhydrides of these acids. These form the humins of brown coal. Under the influence of pressure and a higher temperature carbon dioxide and methane are given off, and a process occurs resulting in the ultimate formation of bituminous coal, the humins of which are insoluble in sodium hydroxide even on boiling. The humins of " pitch coal " become soluble on boiling with sodium hydroxide, this forming a member of the coal series intermediate between bituminous and brown coal, the humins of the latter being directly soluble in alkali.

A. B. MANNING.

Examination and evaluation of coals for carbonising purposes. G. WEYMAN (Gas J., 1926, 173, 601—603, 677—679, 746—747).—From coal to coal the quantities and qualities of the same products may vary, and even different products may result. The size of the coal affects the time of carbonisation, dust impeding the passage of gases and hence of heat through the charge. Hence hardness is an influential factor. Sieving tests are of little value in this connexion; a better index is the bulk density of a 10-ton truck-load. Ultimate analysis does not yield information of much use, but knowledge of the amount and composition of the ash is of greater value. Extraction with solvents is of little practical value. The calorific value does not indicate how the coal will behave when carbonised, unless it is considered in conjunction with the volatile matter. Proximate analysis gives indefinite and incomplete results. Ordinary methods of assay, in which large-scale practice is imitated, do not give sufficient of each product for thorough examination The performance of large-scale tests generally repays for the effort expended. The best method of test, however, is one which is as small as is compatible with yielding sufficient products for analysis. A plant carbonising 1/1000 ton of ½-in. coal has accordingly been devised. It consists of a metal retort, gas-heated in a muffle furnace under conditions permitting of accurate temperature control. The off-take pipe passes from the side of the retort (so that tar will not reflux and crack) to air- and water-cooled condensers which in turn lead the gas through a water and an acid washer, oxide purifier boxes, and a meter fitted with an attachment for recording the rate of gas evolution, into a holder. The yields and the behaviour of the products when tested in the usual way are ascertained. As a basis of evaluation a standard coal is selected and the yield of each product per ton of coal carbonised is multiplied by its basic price in pence per unit. The values of all the products per ton of coal thus obtained are summated and the value of each calculated as a

percentage of the total. This percentage value for each product, divided by the yield, gives a unit value which is used as a basis to compare subsequent coals with the standard. The total value of all the products will then amount to more or less than 100, a figure indicative of the return to be expected from the coal used. This is gross valuation. Nett valuation is obtained by allowing a 1% difference for 1% difference in the moisture (which, in the standard coal is 2%), a correction for difference in ash (standard, 8%) at twice this rate, and a further correction for rate of carbonisation as measured by average rate of gas evolution. R. A. A. TAYLOR.

Path of the gases in the distillation of coal. T. P. L. PETIT (Het Gas, 1926, **46**, 50—51; Chem. Zentr., 1926, I, 2161).—It is now generally accepted that as the coking of the coal in the oven proceeds from the wall towards the centre the gases evolved escape through the pores of the coke already formed in the direction of the nearer oven wall, and not, as earlier theories have stated, through the coal core. According to Hilgenstock (Gas- u. Wasserfach, **49**, 617) the gases at first pass through the coal core until this is cemented together by the tar produced, then, with the advance of the coked layer, the tar is gradually gasified and the gases escape again through the core. The author has already refuted Hilgenstock's theory (Polytechn. Weekblad, 1921, Nr. 1) on the basis of the results of Teune and Kummel (Het Gas, **40**, 168). A simple apparatus is now described which provides indisputable proof that the evolved gases pass exclusively through the pores of the coked layer. A. B. MANNING.

Behaviour of a coal of recent formation on prolonged heating at 110°. D. J. W. KREULEN (Chem. Weekblad, 1926, **23**, 186—187).—After the first hour, the coal increased in weight, the increase continuing but becoming gradually slower for 1275 hrs., after which an intermediate period supervened, followed by a diminution of weight. S. I. LEVY.

Analysis of mixtures of unsaturated hydrocarbon gases. Determination of erythrene in gas mixtures. A. DOBRJANSKI (Neftianoe i slancevoe Chozjajstvo, 1925, **9**, 565—577; Chem. Zentr., 1926, I., 2220—2221).—A method for the determination of the proportions of different olefines in gas mixtures depends upon the varying rates of absorption of members of the olefine series by sulphuric acid of different concentrations. Mixtures containing the gases were treated with sulphuric acid in a glass cylinder of 19·9 sq. cm. cross section, the diminution in volume being measured in the cylinder to avoid transference from one vessel to another and to ensure exposure of the gas to a constant surface of the acid. The rates of absorption of the olefines by sulphuric acid of different strengths are approximately as follows:—ethylene, 0·7 c.c. and 0·007 c.c. per min. by 101·2% and 87·87% acid; propylene, 2·2 c.c. and 0·009 c.c. per min. by 85% and 67·2% acid; *iso*butylene, 3·0 c.c. and 0·013 c.c. per min. by 67·2% and 22·3% acid, respectively. The proportions of these gases in mixtures are determined by bringing the gas successively in contact with 63—64% acid, which absorbs practically nothing but the *iso*butylene, with 83—84% acid, which absorbs the propylene, and with 100—102% acid, which absorbs the ethylene. In a modified procedure for use when the mixture contains erythrene, *iso*butylene is absorbed by 45—46% acid, propylene and erythrene together by 8?% acid, and ethylene by weak oleum; a separate sample of the gas is then shaken with bromine water, formed *in situ* by the addition of potassium bromate and potassium bromide to sulphur dioxide solution in the reaction vessel, and excess bromine is titrated with sodium thiosulphate in the presence of potassium iodide and starch. Erythrene absorbs 4 atoms and *iso*butylene only 2 atoms of bromine under these conditions. L. A. COLES.

Composition of Swedish generator shale oils. G. HELLSING and J. S. W. TROEDSSON (Ark. Kem. Min. Geol., 1925, **9**, [22], 1—12; Chem. Zentr., 1926, I., 2064).—The oil obtained from a Swedish shale used as fuel in a generator is considerably richer in aromatic hydrocarbons than the oil obtained by the usual dry distillation of the shale (cf. B., 1921, 536 A). It is also rich in dissolved sulphur. The latter acts as a dehydrogenating agent at high temperatures, increasing the content of aromatic hydrocarbons at the cost of the naphthenes and other substances present. Both crude oils are probably similar in composition to begin with, their difference arising only from the difference in the distillation process employed. One of the principal constituents of the fraction 155—185° of the generator oil is ψ-cumene, which was isolated as the sulphonamide. The residue after sulphonation consists of a mixture of naphthenes ($C_{10}H_{20}$ and $C_{11}H_{22}$), as was verified by nitration. The presence of β-decanaphthene in the residue was shown by bromination. According to Bruhn (Chem.-Ztg., 1898, 900) the relatively high content of aromatic hydrocarbons in Swedish shale oil is explained first by the formation of naphthenes from oleic acid (set free from esters present in the oil), by loss of carbon dioxide, and rupture at the double bond, and then by their dehydrogenation due to the action of the sulphur at high temperatures. A. B. MANNING.

Examination of spirit produced by the hydrogenation of coal. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1926, **12**, 77—78).—The spirit examined was derived by the "Berginisation" of Orgreave Washed Slack. By a long series of fractionations, observations of refractive indices and iodine values, and preparation of nitro-derivatives of the aromatic compounds present, the authors arrive at the following composition for the spirit:—Unsaturated compounds, 3·1%; benzene, 2·7; toluene, 1·9; xylenes etc., 2·9; butanes, *iso*pentanes, etc., 10·8; *n*-pentane, 10·5; *iso*hexane etc., 6·3; *n*-hexane, 10·0; *iso*heptane etc., 2·9; *n*-heptane, 6·5; *cyclo*pentane, 0·8; methyl*cyclo*pentane, 4·5; *cyclo*hexane, 10·3; methyl*cyclo*hexane, 11·4; residue, 15·4% by weight. The spirit examined

formed approximately 10% of the weight of the coal treated. W. N. Hoyte.

Action of sulphuric acid on petrol. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1926, **12**, 68—76).—The authors treated two petrols with sulphuric acid in a series of washes, the strength of the acid being steadily increased at each wash, from 79·1% to 100·5%. Determinations of percentage loss of weight, density, refractive index, and aniline point, following this treatment indicated that polymerisation and removal of unsaturated compounds take place with acid of strength below 88%, and that with acid above that strength removal of aromatic compounds takes place. W. N. Hoyte.

Symposium on determination of unsaturated hydrocarbons (J. Inst. Petrol. Tech., 1926, **12**, 48—67).—J. S. S. Brame critically reviewed current methods, and suggested the use of 84·5% sulphuric acid for the removal and polymerisation of unsaturated compounds.

A. E. Dunstan pointed out that the assumption that iodine values referred exclusively to unsaturated compounds was probably incorrect.

E. L. Lomax and E. S. Pemberton showed that treatment with 80% sulphuric acid is inadequate for the complete removal of olefines and advised the splitting of the spirit to be examined into separate fractions by distillation prior to treatment with acid.

H. Moore described experiments using various strengths of sulphuric acid from 80% to 95% and stated that unsaturated compounds were not removed sufficiently completely by an acid weaker than 90%. A rise in the aniline test of 1° corresponded to a loss by acid absorption of 2·59% of unsaturated compounds.

F. H. Garner stated that under certain conditions combination between unsaturated compounds and aromatic compounds occurs in the presence of 93% sulphuric acid. W. N. Hoyte.

Vapour pressure of [motor] fuel mixtures. II. J. S. Lewis (J. Inst. Petrol. Tech., 1926, **12**, 32—47; cf. B., 1925, 487).—The vapour pressures of the mixtures, alcohol-*cyclo*hexane, alcohol-hexane, alcohol-blend of hexane and heptane, benzene-*cyclo*hexane-alcohol, at temperatures between 20° and 50°, have been studied. Experiments with three motor spirits having equal vapour pressures, showed that the greatest increase in vapour pressure occurred on addition of alcohol to the paraffinoid spirit; a smaller increase occured on adding alcohol to the naphthenic spirit; and the least increase when the benzol spirit was used. Tables are given showing the vapour pressure at temperatures between 20° and 50° of the ternary systems alcohol–*cyclo*hexane–water and alcohol–*cyclo*hexane–benzene and the boiling points of all the possible constant-boiling point mixtures, binary, ternary, and quaternary, of the components alcohol, benzene, *cyclo*hexane, and water. In blending hydrocarbons of the paraffin series the vapour pressure can be calculated approximately from the mixture formula. The actual value of the vapour pressure is higher than the calculated value and the difference increases with increasing differences between the vapour pressures of the components. The difference between the actual and calculated values is greater when a naphthene and still greater when an aromatic hydrocarbon is dissolved in a paraffin. When naphthene and aromatic hydrocarbons are mixed the actual value far exceeds the calculated value and in the case of benzene and *cyclo*hexane a mixture of maximum vapour pressure is obtainable. W. N. Hoyte.

Effect of water on aniline points [of hydrocarbons]. W. R. Ormandy and E. C. Craven (J. Inst. Petrol. Tech., 1926, **12**, 89—91).—The water content of an approximately dry sample of aniline was determined from observations on the freezing point of the sample. Using this sample as starting point, aniline of known water content, varying from 0·07% to 1·75%, was used for the determination of the aniline point of heptane, *cyclo*hexane, and a commercial motor spirit. The authors find that 1% of water in the aniline causes a rise in aniline point of 6·2° for heptane and the spirit examined and 5·6° for *cyclo*hexane. They suggest the use of a similar method for determining the water content of petroleum spirit. W. N. Hoyte.

See also pages 399, **Determination of carbon disulphide and hydrogen sulphide** (Hegel). 404, **Ammonia synthesis from coke-oven gas** (Claude); **Ammonium sulphate** (Bateman). 405, **Determination of carbon monoxide in hydrogen** (Schuftan). 410, **Sulphur in producer-gas** (Brown).

Patents.

Artificial fuel. L. L. Summers (U.S.P. 1,576,253, 9.3.26, Appl., 29.9.21).—Raw coal is extracted with a solvent, and the carbonaceous residue, after compression and distillation, is covered with the extract. W. T. K. Braunholtz.

Pulverisation of coal. H. Kreisinger, Assr. to Combustion Engineering Corp. (U.S.P. 1,576,335, 9.3.26. Appl., 16.1.25).—Coal is pulverised in a mill in a system including a separator and air as carrier, and is preheated to such a degree that its sensible heat suffices to dry the coal during pulverisation in the presence of the carrying air. W. T. K. Braunholtz.

Carbonisation of coal. Illingworth Carbonization Co., Ltd., and S. R. Illingworth (E.P. 248,077, 27.11.24).—A succession of blows are struck by means of hammers or by the coal-feeding tubes on the upper part of a vertical retort during carbonisation. The hammers can be connected with the coal feed valve in order to control the rate of striking. A coal with 22% of volatile matter carbonised at 600° produced a fuel of apparent density 1·09 when the retort was vibrated as compared with 0·832 with no vibration. A. C. Monkhouse.

Cooling of coke. Illingworth Carbonization Co., Ltd., and S. R. Illingworth (E.P. 248,078, 27.11.24).—The sensible heat of the coke is used to dry the raw coal which is to be carbonised. The

coke is discharged into a cooling wagon containing a measured amount of non-caking coal and the wagon is rotated; caking coal is then added and after further mixing the contents of the wagon pass to the coke cutter and are then discharged on to a screen where the smaller coal and breeze pass into a hopper and thence to the disintegrator and retort hoppers, whilst the coke is conveyed to the coke store. The heat in low-temperature coke is sufficient to dry the corresponding charge of coal from 10% to 3% of moisture. The proportion of caking to non-caking coal can be adjusted and the process made continuous by combining with the cooler a conveyor working in a closed chamber beneath the retorts. A. C. MONKHOUSE.

Destructive distillation [of maize cobs etc.]. F. C. ATKINSON, Assr. to J. W. GARLAND, INC. (U.S.P. 1,572,510, 9.2.26. Appl., 20.8.19).—Maize cobs or like waste material are crushed, extracted with water at a pressure of 3 atm. for 3 hrs., dried, and distilled out of contact with air to 440°. Charcoal of good quality free from mineral matter is obtained as a residue. The distillate separates into two layers on cooling, the upper being an aqueous layer containing acid substances. The lower oily layer is fractionated when cresols distil at 150—250° and are purified by treatment with sodium hydroxide solution. A creosote similar to beech wood creosote is obtained. T. S. WHEELER.

Decolorising and defecating product. G. J. ESSELEN, JUN., Assr. to UNITED FRUIT CO. (U.S.P. 1,575,561, 2.3.26. Appl., 6.10.22).—The product consists of a charcoal derived principally from the pith of the sugar-cane, intimately associated with an alkaline material. R. A. A. TAYLOR.

Oven for the distillation of solid material. O. LASCHE and C. GEISSEN, Assrs. to KOHLENVEREDLUNG G.M.B.H. (U.S.P. 1,576,916, 16.3.26. Appl., 6.3.35).—The oven comprises an outer cylindrical shell and an inner, thick-walled, hollow body of refractory material pierced with holes. There is an annular space between the shell and inner body, and heating gas is introduced axially into the latter. W. T. K. BRAUNHOLTZ.

Manufacture of water-gas. G. PATART (F.P. 599,589, 25.5.25).—A mixture of oxygen, steam, and pulverised fuel is blown into a generator of the usual type. A. B. MANNING.

Flameless combustion of vapours and gases by means of catalysts. H. SCHIMMEL (G.P. 424,209, 7.10.24).—The vapours or gases are forced under pressure in extremely fine streams through the catalyst, whereby they are completely burnt. A. B. MANNING.

Using low-compression fuels [in internal-combustion engines]. T. MIDGLEY, JUN., Assr. to GEN. MOTORS CORP. (U.S.P. 1,575,436–444, 2.3.26. Appl., 18.9.23).—A fuel which detonates at low compressions in internal-combustion engines is mixed with a volatile compound of selenium tellurium, arsenic, antimony, phosphorus, cadmium, bismuth, titanium, or tin, in order to diminish the tendency to detonate. W. N. HOYTE.

Treating oil-bearing shales. N. H. FREEMAN (E.P. 248,014, 4.9.24).—The finely crushed shale mixed with lime is subjected to a temperature of about 180° for 1 hour; by this means the shale oil is rendered soluble. This is followed by extraction of the treated shale with kerosene, not volatile below 150°. The shale is separated from the oil either by a centrifuge or a filter. The shale oil is then separated by distillation from the kerosene, which is used again. It is claimed that the addition of lime preliminary to the heat treatment lowers the sulphur content of the resulting oil. W. N HOYTE.

Production of light hydrocarbons. ALLGEM. GES. FÜR CHEM. IND. (E.P. 244,697, 6.5.25. Conv., 19.12.24).—A liquid catalyst is prepared by the action of anhydrous aluminium chloride at 30—90° on a purified oil of high boiling-point such as gas oil. The purification is effected by washing the oil with liquid sulphur dioxide, or methyl, ethyl, or amyl alcohol, or by treatment with sulphuric acid or silica gel. About 10% by volume of the catalyst is introduced into the cracking stock maintained at a suitable temperature (about 200°). Cracking proceeds smoothly, and the process is characterised by the small quantity of gas and the large proportion of saturated hydrocarbons formed. W. N. HOYTE.

Cracking of liquid hydrocarbons. V. L. OIL PROCESSES, LTD., O. D. LUCAS, and E. L. LOMAX (E.P. 248,115, 9.12.24).—The process is based on the observation that the percentage of cracked spirit obtainable in one operation is increased by maintaining a low concentration of lighter oils, produced by cracking, in the residues. The oil is preheated and pumped under a suitable pressure through a pipe still where it is heated to cracking temperature. It passes to a reaction vessel maintained at cracking temperature. The lighter oils produced pass to a refluxing chamber situated above the reaction chamber and maintained at a temperature at least 200° lower than that of the latter chamber. The condensed oil from this chamber passes back to the reaction chamber, either being sprayed in at the top or introduced at the bottom with the main charge. The vapours from the reflux chamber pass through an expansion chamber to a dephlegmator. The vapours from the dephlegmator pass via a vapour refining apparatus to the main condenser. The liquid deposited in the expansion chamber and dephlegmator runs through the preheater above mentioned to the residue tanks. The liquid residue from the reaction chamber passes through coils in the base of the dephlegmator to the expansion chamber. Suggested temperatures and pressures are as follows:—Oil exit from pipe still, 600°; reaction chamber, 450—550° and 600—1000 lb. per sq. in.; refluxing chamber, 360—400° and 600—1000 lb. per sq. in.; expansion chamber, 75 lb. per sq. in. and 350°; dephlegmator, 75 lb.

per sq. in.; outlet for gas and vapour from the column, 25 lb. per sq. in. W. N. Hoyte.

Fractional distillation of hydrocarbons. C. de Ganahl and F. C. Koch (E.P. 248,186, 16.3.25).—The apparatus comprises substantially a pipe still and a dephlegmator. When sharp fractionation is desired the preheated charge enters the dephlegmator at a suitable point, the light fractions are removed, the residue is pumped through the pipe still into a vaporising chamber, and the vapours from this pass up the dephlegmator where they undergo fractionation. The vapours from the top of the dephlegmator pass via the preheater mentioned above through a condenser, thence a portion of the condensate is run to stock, and the remainder is pumped to the top of the dephlegmator to assist fractionation. The liquid deposited in the vaporising chamber is pumped to a second unit similar to the one described, and is introduced as before into the dephlegmator at a suitable point. Sufficient units are used to give the degree of fractionation desired. When only rough fractionation is required the preheated charge is pumped direct to the pipe still instead of being introduced into the dephlegmator. W. N. Hoyte.

Operation of oil stills. J. E. Bell, Assr. to Sinclair Refining Co. (U.S.P. 1,574,547, 23.2.26. Appl., 10.8.23; cf. E.P. 240,355; B., 1926, 120).—Cracking stills are heated by hot products of combustion from a furnace and a portion of the hot waste gases is passed again over the oil heating surfaces, in admixture with fresh hot gases from the burners. W. N. Hoyte.

Pressure still for oil refining. D. Pyzel, Assr. to Simplex Refining Co. (U.S.P. 1,574,935, 2.3.26. Appl., 13.3.22).—In a steel pressure still for cracking oil a number of thermocouples are provided to indicate the temperature of various portions of the still. Any deposition of carbon causes local overheating owing to its insulating properties, and this is shown by the corresponding thermocouple. The still is then cooled for cleansing purposes. T. S. Wheeler.

Cracking oils under pressure. J. E. Bell, Assr. to Sinclair Refining Co. (U.S.P. 1,575,031, 2.3.26. Appl., 1.4.24).—The oil is raised to cracking temperature under pressure, the resulting vapours are condensed fractionally under pressure, and the amount of condensation is increased or decreased with increase or decrease of pressure. Uncondensed vapours escape through a fixed orifice and are condensed at atmospheric pressure. W. N. Hoyte.

Apparatus for condensing [hydrocarbon] vapours. E. W. Isom and J. E. Bell, Assrs. to Sinclair Refining Co. (U.S.P. 1,575,269, 2.3.26. Appl., 27.5.21).—A vessel fitted with cooling coils is kept about half full of the liquid condensate, and the mixture of hydrocarbon vapours and gases under treatment is introduced through nozzles situated below the liquid level so as thoroughly to agitate the liquid. W. N. Hoyte.

Securing temporary intimate contact [without emulsification] between immiscible liquids. L. D. Jones, Assr. to Sharples Specialty Co. (U.S.P. 1,575,116, 2.3.26. Appl., 3.9.21).—An apparatus for causing intimate contact between immiscible liquids without emulsification, *e.g.*, for treating viscous lubricating oils with sulphuric acid or alkali-refined oils with water, consists of a rotating vertical cylinder to the bottom of which the interacting liquids are introduced. A number of superposed horizontal discs arranged centrally in the cylinder may be used, so that as the liquids rise in the cylinder the lighter passes in a thin film within the heavier around the edges of the discs. Rings may be fitted to the inside of the cylinder, or a combination of peripheral rings and central discs may be used. At the top of the cylinder the liquids are centrifugally separated and withdrawn. T. S. Wheeler.

Refining and decolorising bitumens. G. W. Acheson (U.S.P. 1,574,742, 2.3.26. Appl., 8.10.24).—The bitumen is treated with a solid reflocculated absorbent material (cf. U.S.P. 1,563,713, B. 1926, 224) in conjunction with an acid reagent. The refined bitumen is separated from the sludge and is then treated with an anhydrous oxide to decolorise it. W. N. Hoyte.

Method of treating particles of matter [coal]. B. E. Eldred and R. N. Graham, Assrs. to Nat. Carbon Co. (U.S.P. 1,578,274, 30.3.26. Appl., 10.3.22).—See E.P. 199,753; B., 1923, 812 a.

Production of coke and tar from coal. F. Seidenschnur (U.S.P. 1,578,376, 30.3.26. Appl., 2.10.24).—See E.P. 228,494; B., 1925, 700.

Gas off-take pipes for fuel distillation retorts or coke ovens. W. Mueller (E.P. 245,728, 12.10.25. Conv., 8.1.25).

Sulphur from gases containing hydrogen sulphide (G.P. 423,395).—See VII.

Bituminous emulsions (E.P. 248,859).—See IX.

III.—TAR AND TAR PRODUCTS.

Patent.

Fractional distillation of hydrocarbons (E.P. 248,186).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Vat dyes and some recent developments. R. F. Thomson (J. Soc. Dyers and Col., 1926, **42**, 124—126).

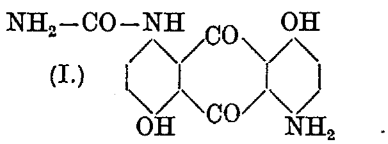
See also A., April, 403, **Colour and chemical constitution** (Moir). 407, **Nitro-derivatives of 1-hydroxy-3-methylanthraquinone and related products** (Eder and Manoukian). 408, **Anthraquinonylamides of substituted anthraquinone-carboxylic acids** (Rollett). 412, **Benzene-4-hydrazo- and -azo-pyridine derivatives** (Koenigs

and others). 415, **Synthesis of substitution derivatives of indigotin** (OVERMYER). 420, **Determination of nitro-compounds** (KOLTHOFF and ROBINSON).

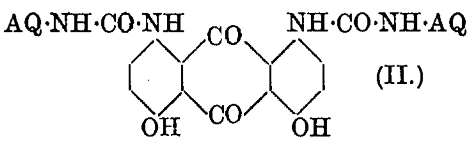
PATENTS.

Manufacture of new anthraquinone dyestuffs possessing affinity for acetyl [cellulose acetate] silk. BRIT. DYESTUFFS CORP., LTD., W. H. PERKIN, and C. HOLLINS (E.P. 248,858, 16.12.24).—New carbamides of the anthraquinone series containing two or three anthraquinonyl groups linked together by the chain —NH·CO·NH—, and having affinity for cellulose acetate silk, are obtained by condensing α-anthraquinonylethyl carbamate or α-anthraquinonylcarbamic chloride with equimolecular proportions of α-aminoanthraquinones or derivatives, preferably those having two amino-groups in α-positions, such as 1 : 4-, 1 : 5-, or 1 : 8-diamino-anthraquinones, diaminoanthrarufin, or diaminochrysazin. For example, 13·5 kg. of diaminoanthrarufin when heated with 14·5 kg. of α-anthraquinonylethyl carbamate (or an equivalent proportion of the corresponding carbamic chloride) at the m.p. of the mixture for about ½ hr., form a product which dyes cellulose acetate silk a brown shade and is probably the monocarbamide (I.) :—

(I.)



(II.)



(AQ=an anthraquinonyl residue).

Diaminochrysazin and α-anthraquinonylethyl carbamate condensed in the same way give a product which is probably the dicarbamide (II.) and which gives a steel-grey shade. A. COULTHARD.

Manufacture of anthraquinone derivatives. H. DODD, W. C. SPRENT, and UNITED ALKALI CO., LTD. (E.P. 248,874, 29.12.24).—Iron and nickel have a specific action in directing the condensation of aromatic amines with quinizarin. In an iron vessel the simple condensation product is obtained free from the double one, whilst in a nickel vessel the double condensation takes place if iron is absent. For example, 60 g. of quinizarin, 170 g. of dry *p*-chloroaniline, and 30 g. of boric acid are stirred and gently heated in an iron vessel. After gradually raising the temperature to 130° for about 2 hrs., the excess of *p*-chloroaniline is removed by dilute hydrochloric acid. The residue is the simple condensation product, $C_{14}H_6O_2(OH)\cdot NH\cdot C_6H_4Cl$, in 80% yield. 60 g. of quinizarin, 200 g. of *p*-chloroaniline, and 30 g. of boric acid, heated for 2 hrs. in a nickel vessel at a temperature not above 150°, give a yield of 81 g. of the double condensation product as a blue powder. The two dyes after sulphonation give totally different colours on wool.
A. COULTHARD.

Manufacture of dyestuff intermediates [2-chloroanthraquinone]. J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 248,411, 8.9.24).—2-Chloroanthraquinone made by heating *p*-chlorobenzoylbenzoic acid with sulphuric acid of 85—90% strength is only slightly soluble in the residual acid and crystallises out on cooling. The acid filtrate may be revivified by the addition of oleum or sulphur trioxide and used repeatedly. For example, 100 pts. (by weight) of *p*-chlorobenzoylbenzoic acid are dissolved in 1000 pts. of 90% sulphuric acid. The mixture is well stirred and heated to about 170°, kept at this temperature for about 1 hr., and then cooled. The 2-chloroanthraquinone crystals are filtered off and after washing and drying have m.p. 206—208°. 850 pts. of the acid filtrate are then mixed with 150 pts. of 65% oleum and the process is repeated, and so on, until the accumulation of impurities renders the filtrates unfit for further use.
A. COULTHARD.

Preparation of aminodianthrimides. BRIT. DYESTUFFS CORP., LTD., H. M. BUNBURY, and R. ROBINSON (E.P. 248,866, 19.12.24).—Anthraquinonemono-oxamic acids (obtained in good yields direct from diaminoanthraquinones and oxalic acid) of the type $NH_2\cdot AQ\cdot NH\cdot CO\cdot CO_2H$, where AQ=a bivalent anthraquinone residue, condense with halogenated anthraquinones to form mono- and dianthrimides. During the condensation the oxalyl group is converted wholly or partly into the formyl group by loss of carbon dioxide. Hydrolysis of the dianthrimide derivative removes the oxalyl (or formyl) group, leaving an aminodianthrimide. For example, 4 : 5′-diamino-1 : 1′-dianthrimide is prepared by heating 11·4 pts. of 4-benzamido-1-chloroanthraquinone, 10 pts. of 1 : 5-diaminoanthraquinonemono-oxamic acid, 3 pts. of fused sodium acetate, 0·3 pt. of copper powder, and 100 pts. of nitrobenzene for 15 hrs. at a gentle boil. The 5 : 5′- and 4 : 5′-diamino-1 : 1′-dianthrimides are prepared in a similar way. They are valuable intermediates for vat dyestuffs. A. COULTHARD.

Soluble products from vat dyes (E.P. 247,787 and 248,802).—See VI.

Phenolphthalein (U.S.P. 1,574,934).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

[Swelling] action of caustic soda on cellulose pulp. J. D'ANS and A. JÄGER (Kunstseide, 1925, 7, 252—256).—The swelling action of caustic alkalis in the presence or absence of neutral salts on bleached and unbleached soda- and sulphite-celluloses was determined by observation of the increase in weight of the cellulose in the form of paper after immersion in the alkaline solutions and drainage from excess of liquor, correction being made for the density of the liquor. The maximum swelling effect was

produced by solutions containing 11—12% of caustic soda and 15% (wt./vol.) of caustic potash, the swellings being about 11% and 5·5% respectively. Swelling is considerably reduced by the presence of neutral salts and other substances, *e.g.*, sodium chloride, sodium carbonate, ammonia, alcohol, sugar, tannic acid, and gelatin. For example, a 12% solution of caustic soda containing 10% of sodium chloride produces a swelling of only 4·9%. Swelling is also reduced by rise of temperature of the caustic liquor, but this effect is less evident with highly concentrated alkaline solutions. For example, a 10% solution of caustic soda produces swellings of 14·6 and 6·6% at temperatures of 13—16° and 23—25° respectively, whereas under similar conditions the swellings produced by an 18% solution of caustic soda are 6·6% and 5·9%. Dry cellulose liberates 2·6 cal. per g. when moistened with 6% of water. Over a period of 20 hrs. alkali-cellulose saturated with 17% caustic soda liberates 0·417 cal. per hr. per g., whereas under similar conditions during 24 hrs. dry alkali-cellulose (as prepared for the manufacture of artificial silk) liberates 0·27 cal. per hr. per g., the rate of liberation of heat not being affected by the presence of oxygen. Alkali-cellulose heated to 60° and then cooled liberates no heat on keeping (cf. B., 1925, 984). A. J. Hall.

Determination of the degree of swelling of cellulose by Schwalbe's hydrolysis number method. G. Bernardy (Z. angew. Chem., 1926, **39**, 259—261).—As usually carried out this method (Schwalbe, B., 1910, 689) yields irregular results. Reproducible results are obtained if care is taken in neutralising the acid after the first (hydrolytic) boil. An excess of even a few drops of the concentrated sodium hydroxide results in the cuprous oxide, subsequently produced by reduction of the Fehling's solution, being brownish-yellow instead of red, and in such cases high results are obtained. The apparatus is modified to facilitate exact neutralisation. A. Geake.

Scientific foundations of the viscose process. E. Heuser and M. Schuster (Cellulosechem., 1926, **7**, 17—55).—The chemical mechanism of the process of ripening of viscose as originally established by Cross and Bevan is upheld, and the data on which recent investigators have based a purely colloidal explanation are shown to be unreliable owing to imperfect purification of the xanthates examined. Viscose is the sodium dithiocarbonic ester of cellulose or alkali-cellulose. Preparations precipitated by alcohol contain variable amounts of sodium hydroxide depending on the conditions of dilution; xanthates precipitated and purified with sodium chloride are free from sodium hydroxide. Ripening is simply a process of hydrolysis of the ester with elimination of sodium hydroxide and carbon disulphide, these products then undergoing secondary reactions, principally with formation of sodium trithiocarbonate. The primary product of complete xanthogenation is the C_{12} xanthate in the ratio $(C_6H_{10}O_5)_2 : CS_2 : NaOH$; no evidence could be obtained of the formation of a C_6 xanthate nor of any higher degree of xanthogenation. When the proportion of carbon disulphide is restricted to the amount theoretically necessary to produce the C_{12} xanthate (23·6% of the cellulose), there is difficulty in preparing a homogeneous solution and the stability of the viscose does not become normal until the C_{24} stage has been reached. A properly soluble viscose is not obtained unless the concentration of the mercerising sodium hydroxide has been sufficient to produce the alkali-cellulose compound $(C_6H_{10}O_5)_2,NaOH$; in practice a slightly higher concentration is desirable. The same law holds in the case of the hydroxides of the other alkali metals, lithium, potassium, and rubidium, all of which give "viscoses" if employed at equivalent concentrations. The solvent powers of *N*-solutions of the hydroxides of the alkali metals decrease as the atomic weights increase. For comparative purposes the products of xanthogenation may conveniently be dissolved in *N*-sodium hydroxide. For instance, a rubidium xanthate gave an imperfect viscose when dissolved in *N*-rubidium hydroxide but a perfectly smooth viscose when dissolved in sodium hydroxide; the latter viscose, however, yielded a pure rubidium xanthate when neutralised and precipitated by salt. Viscose solutions are most stable against coagulation when they contain 8% of free sodium hydroxide; this also corresponds with the condition of minimum viscosity. The effect of by-products (salts) and of higher concentrations of sodium hydroxide is to hasten coagulation. The complexity of the cellulose molecule, or its degree of dispersion, as measured by the viscosity of the regenerated product in cuprammonium, undergoes a quick change after the immersion in the mercerising liquid, the viscosity falling in 6 hrs. from 75 to 25. During the ageing of the alkali-cellulose the fall in viscosity proceeds more slowly to 12·3 after 3 days. During the xanthate reaction another quick drop in viscosity to 8·3 is recorded, after which, during the ripening of the viscose, very little change in the cellulose complex can be detected. The changes in the viscosity of the viscose itself are initially influenced by colloidal physical conditions. The dispersion of the cellulose xanthate requires an appreciable time or vigorous mechanical treatment. The viscosity of freshly prepared viscose is fairly high and falls, as dispersion becomes more complete, to a minimum after 24—48 hrs. Subsequently there is a steady rise in viscosity during ripening, attributable to loss of ester groups from the xanthate and accumulation of saline by-products in the solvent; the rise in viscosity immediately before coagulation is very rapid. J. F. Briggs.

Determination of carbon disulphide and hydrogen sulphide in gaseous mixtures [from decomposition of viscose] K. Hegel (Z. angew. Chem., 1926, **39**, 431).—The mixture of gases, containing carbon disulphide and hydrogen sulphide, evolved when viscose (10 g.) is heated with 50 c.c. of *N*-sulphuric acid, is swept by a stream of nitrogen through calcium chloride at 55°, and then through a freshly prepared, dry, fat-free, 1% ether solution of

triethylphosphine cooled to —10°. Carbon disulphide is completely absorbed by the phosphine with formation of the double compound, Et_3P,CS_2, which is precipitated quantitatively from ether solutions of triethylphosphine. The crystalline compound is dried at room temperature *in vacuo* before weighing. Hydrogen sulphide is determined by passing the gas mixture through 0·1*N*-iodine solution, the excess of which is titrated with thiosulphate.

L. M. CLARK.

Naphthenic acid esters of cellulose. G. KITA, T. MAZUME, T. NAKASHIMA, and I. SAKURADA (Cellulose Ind., Tokyo, 1926, 2, 31—38).—Naphthenic acid fractions having acid values between 199 and 250, and prepared free from hydrocarbons by suitable treatment of petroleum fractions, were used in the forms of acid anhydrides and acid chlorides (cf. B., 1926, 45). Cellulose esters are obtained with the naphthenic acid anhydrides but the degree of esterification is low. With the acid chlorides in the presence of pyridine, esters containing up to 2·5 mols. of naphthenic acid to 1 mol. of cellulose are produced; unmodified cellulose reacts less readily than hydrocellulose. The esters resemble those produced with the higher fatty acids; those with high naphthenic acid content show definite melting points. Naphthenic esters of unmodified cellulose are substantially insoluble, although a portion is soluble in benzene; the solubility depends not only on the naphthenic acid content but also on the condition of the cellulose. The di-naphthenic ester prepared from hydrocellulose is completely soluble in benzene, chloroform, carbon tetrachloride, and ether. The originally insoluble ester also becomes soluble in these media after fusion. Naphthenic esters containing up to 1 mol. of naphthenic acid to 4 mols. of cellulose are obtained by treating alkali-cellulose with the acid chlorides. J. F. BRIGGS.

Production of straw-board pulp. J. D. RUE and W. MOUSSON (Paper Trade J., 1925, 81, [15], 52—53, [20], 49; Papier-Fabr., 1926, 24, 169—172). —Pulp made from straw boiled with milk of lime imparts an undesirable odour to eggs packed in it. A product with a much milder odour is obtained by boiling with sodium sulphite, with the addition of sodium carbonate to prevent corrosion of the boiler. The proportions recommended are 1·5 lb. of sodium sulphite, 7·5 lb. of sodium carbonate, and 20 galls. of water to 134 lb. of straw. The temperature is raised to 145° during 2 hrs., and is maintained for 6 hrs. The product contains 80% of cellulose, instead of 70% when lime is used. The cost of chemicals is greater but may be partly or wholly compensated by increased yield and better quality of product. A. GEAKE.

Removing printers' ink from old paper. K. STEPHAN (Z. angew. Chem., 1926, 39, 269—270). —The process of Berl and Pfannmüller (B., 1926, 286) is stated to be inefficient and too expensive.

A. GEAKE.

Tall oil, a by-product of sulphate-cellulose manufacture (DITTMER).—See XII.

PATENTS.

Manufacture of artificial silk. E. LUNGE, and COURTAULDS, LTD. (E.P. 248,043, 20.11.24).—In order to control the ripening of viscose the pipes and pumps conveying the solution from the chambers in which it is prepared to the spinning nozzles, are immersed in baths the temperature of which is controlled. To prevent corrosion of the pumps and hardening of viscose adhering to their moving parts, the bath surrounding them contains, preferably, a 1% solution of sodium hydroxide or carbonate. This is covered with a layer of oil to prevent absorption of sulphur fumes, which would lead to corrosion. At any stoppage, such as week-ends, the viscose may be cooled to such a temperature that ageing practically ceases. A. GEAKE.

Filters [for viscose]. E. LUNGE, and COURTAULDS, LTD. (E.P. 248,044, 20.11.24).—The filtering medium covers the top of a long trough and the filtered liquid is received in a similar trough inverted over the filter. The feed and delivery pipes are at opposite ends of the pair of troughs. The filter is designed especially for viscous liquids, such as viscose; it secures a large filtering surface, all of which is equally and effectively used, and avoids the possibility of stagnation at any part. This latter is inportant in the case of viscose since stagnant liquid will ripen, and the product will then lack uniformity. In the preferred form of construction the troughs consist of channels in two discs bolted together; the channels are zig-zagged so as to be as long as possible, and arranged so as to register throughout their lengths. Either end of the one channel may be connected to the feed pipe, and the corresponding opposite end of the other channel is simultaneously opened to the delivery pipe; the possibility of stagnation is thus further reduced. The filtering medium is a suitable fabric or other material, and may be supported by a perforated plate. A. GEAKE.

Manufacture of new cellulose compounds [thiourethanes]. L. LILIENFELD (E.P. 248,246, 9.7.25. Conv., 30.5.25. Addn. to 231,801, cf. B., 1925, 955).—Instead of the cellulose xantho-fatty acid derivatives mentioned in the original specification, other compounds of cellulose containing the CSS-group may be brought into reaction with a primary or secondary amine to form new compounds of *N*-substituted thiourethane type. For instance, cellulose xanthate (viscose) or cellulose xanthic acid, or the products which are obtainable from them by the action of oxidising agents, such as iodine, copper salts, potassium ferricyanide, and the like, or products of the reaction between esters of chlorocarbonic acid and cellulose xanthate or cellulose xanthic acid, or the esters of cellulose xanthic acid (*e.g.*, compounds prepared by the action of alkyl sulphates or iodides on viscose) are treated with primary or secondary aliphatic or aromatic amines or hydroxy-amines in the manner described in the original patent. J. F. B.

Manufacture of fibres, tape, etc. from viscose. A. Kämpf (U.S.P. 1,575,210, 2.3.26. Appl., 7.5.23). —Products are formed from completely unripened viscose prepared from completely unripened alkali-cellulose by spinning the unripened viscose in an acid bath having a temperature of about 4°, the bath containing no added organic matter or salts other than those unavoidably introduced by the operation of spinning. J. F. B.

Increasing the durability of lacquers, threads, and other products from cellulose. P. Le Play (F.P. 595,208, 16.3.25).—Resins or rubber or rubber latex are incorporated in cellulose solutions. For example, artificial silk threads resistant to tensile strain and moisture are prepared from solutions of nitrocellulose or cellulose acetate containing rubber or from viscose or cuprammonium solutions of cellulose containing rubber latex. A. J. Hall.

[Manufacture of] cellulose xanthate. P. Moro (F.P. 597,336, 31.3.25).—Alkali-cellulose is pressed free from excess of caustic lye, then disintegrated and mixed with a solution of sulphur in carbon disulphide in the usual type of jacketed mixing machine for a prolonged period at 30—40° until a clear solution is obtained; after keeping for 12—24 hrs. the product is filtered and spun into artificial silk. Alternatively, the alkali-cellulose is distributed over sieves arranged in one or more towers through which carbon disulphide containing sulphur is drawn by means of a pump which also communicates with a condenser for the recovery of excess carbon disulphide. A. J. Hall.

Separation of vegetable fibres in the manufacture of cellulose. Chem. Fabr. Griesheim-Elektron (F.P. 599,540, 15.6.25. Conv., 16.6 and 3.7.24).—Raw fibrous materials are subjected for one or several hours' digestion with alkali sulphites in presence of dilute acid (sulphur dioxide) at temperatures not below 100°. A. J. Hall.

Treating bagasse for fibre-making purposes. T. B. Munroe (U.S.P. 1,574,254, 23.2.26. Appl., 7.7.24).—Piled bagasse is subjected to an acetic acid fermentation until the fibre bundles are partially resolved, but without deterioration of the strength of the fibres. The fermented bagasse is mixed with a sufficient quantity of fibres derived from wood to assist the drying of the final board; the mixture is cooked, refined, and separated from the products of the cooking process, a fire-resisting material is incorporated with the pulp, and the mass is formed into a heat-insulating fibre board. J. F. B.

Apparatus for making sulphite liquor. F. J. Babcock, Assr. to Stebbins Engineering & Manuf. Co. (U.S.P. 1,576,705, 16.3.26. Appl., 24.7.24).—In a tower system for making acid sulphite liquor two separate reversible limestone towers and a tank for receiving liquor from either of the towers are combined with means for strengthening the acid liquor in the tank. This consists of a supplementary tank in communication with the first tank for receiving relief gas, and a supplementary limestone tower. Liquor may be circulated through the supplementary tank and tower while gas is supplied to the latter. A. Geake.

Manufacture of safety paper. M. Reinheimer (G.P. 416,302, 13.7.23).—Safety paper is prepared by the introduction of nickeled iron powder into the paper pulp in the hollander. The genuineness of the paper can then readily be tested at any time by an electro-magnetic device. A. B. Manning.

Recovery of soda from solutions [used in paper manufacture]. W. M. Wallace (E.P. 248,864, 18.12.24. Addn. to 217,468).—Complete burning of the calcined ash, issuing from the furnace on a travelling grate as described in the chief patent (B., 1924, 707), is assisted by supplying air from above, in addition to that supplied from below. A. Geake.

Regeneration process for black liquor [from sulphate pulp process]. A. H. White, Assr. to J. E. Alexander and E. G. Goodell (U.S.P. 1,575,473, 2.3.26. Appl., 26.12.24. Renewed 6.11.25). —The black liquor from the sulphate pulp process is concentrated and subjected to destructive distillation; the resulting solid residue is calcined together with calcium carbonate to form a mixture of calcium oxide, sodium carbonate, and sodium sulphate; the calcined residue is caused to react with carbonaceous material at a temperature above 300° but below 900° in the absence of air until the sulphate is reduced; the soluble sodium salts are then recovered by leaching. J. F. B.

Manufacture of chlorinated products from cellulose waste lyes. Königsberger Zellstoff-Fabr. & Chem. Werke Koholyt A.-G. (G.P. 419,815, 11.5.22).—Cellulose waste liquor is chlorinated in an electrolytic cell the anodes of which are constructed of materials which allow of varying overvoltage towards chlorine and oxygen respectively so that chlorination and oxidation may be effected simultaneously. A high degree of chlorination may be effected when electrodes of magnetite are used. Chlorination is preferably carried out at a moderately high temperature, and the anode and cathode are separated by a diaphragm; or the process may be made continuous by causing the electrolyte to flow from the cathode to the anode, the diaphragm being omitted. For example, from sulphite-cellulose waste liquors chlorinated products containing 25·4—22·7% Cl, easily soluble in water with a brown colour and containing no free hydrochloric acid, are obtained by using a solution of sodium chloride at 40—50° as electrolyte, with a magnetite anode, an earthenware diaphragm, and a graphite cathode, or a graphite anode and an iron cathode. A. J. Hall.

Utilisation of gases from cellulose digesters. Metallbank u. Metallurgische Ges. A.-G. (G.P. 423,198, 7.11.23).—The hot gases containing sulphur dioxide are led through an electrical precipitation apparatus whereby their heat value is utilised and

they are freed from organic, particularly aromatic and organic acid impurities, and are rendered suitable for re-use. Successive falls of temperature between the units of the precipitation apparatus allow of the fractional separation of constituents of the impurities. A. J. HALL.

Manufacture of viscose. H. DREYFUS (U.S.P. 1,578,588, 30.3.26. Appl., 28.1.22).—See E.P. 183,882; B., 1922, 748 A.

Dialysing membranes from nitrocellulose (U.S.P. 1,576,523).—See I.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Theory of dyeing on wool and silk. P. PFEIFFER and O. ANGERN (Z. angew. Chem., 1926, **39**, 253—259).—Benzeneazophenol forms crystalline compounds with 1 mol. of phenylalanine and of sarcosine, and benzeneazoresorcinol with 2 mols. of sarcosine. The methyl ether of benzeneazophenol does not form such compounds, and it is concluded that salt-like combination takes place between the amino-group of the amino-acid and the phenolic hydroxy-group. Similar compounds with basic dyes could not be obtained. Sarcosine anhydride (dimethyldiketopiperazine) forms crystalline compounds with 2 mols. of benzeneazophenol, benzeneazoresorcinol, and benzeneazosalicylic acid. Azobenzene and the methyl ether of benzeneazophenol do not form such compounds, and it is probable that molecular combination takes place between the carbonyl oxygen of the amino-acid anhydride and the phenolic hydroxyl. Sarcosine anhydride also combines with *p*-aminoazobenzene, *o*-tolueneazo-*o*-toluidine, *o*-phenylenediamine, *m*-phenylenediamine, and β-naphthylamine, but not with *p*-dimethylaminoazobenzene or dibenzyl-β-naphthylamine. These compounds are generally decomposed by suitable solvents. It is concluded that the dyeing of wool and silk is due, in general, to the formation of molecular compounds, which may, in special cases, be of a salt-like character. If combination takes place only on the surface of the fibre this theory becomes similar to the adsorption theory. A. GEAKE.

Dyes not fast to ironing. R. HALLER (Kolloid-Z., 1926, **38**, 248—253).—An investigation of the nature of the dyeing process when blue and violet benzidine dyes are used on cotton, wool, and acetate silk fibres, with special reference to the reversible colour change (blue to corinth-red) when the dyed cotton is touched with a hot iron. The experiments confirm the view that these dyes form colloidal solutions of different degrees of dispersion, the larger particles colouring cotton fibre blue, and the smaller corinth-red (cf. A., 1921, ii, 28, 576). Solutions with a low degree of dispersion are particularly sensitive to temperature changes or to variations in the medium employed. Thus, in hot dye baths, wool and cotton are dyed red by aqueous solutions, but on cooling the colour on the cotton becomes blue-violet; alcoholic solutions hardly affect wool, but dye cotton a permanent corinth-red. The effect of touching the dyed cotton with heated metal is to increase the degree of dispersion of the dye on the fabric, with change of colour from blue to red. The addition of hydrazine hydrate to Diamine Blue 3R causes a similar change, and the solution will then dye cotton corinth-red. The adsorption of the dyes by fibrous alumina and barium sulphate was also investigated. With 1% Diamine Blue 3R and alumina, a dark blue deposit and a corinth-red solution were obtained; using barium sulphate, selective adsorption was clearly demonstrated. The largest particles, which are colourless, are first deposited, then a layer of smaller violet particles, and lastly a small zone of corinth-red particles. This indicates a fixed relationship between the degree of dispersion of the colouring matter and that of the adsorbent. The surface of the adsorbent plays a decisive part also when fibres are used, for swelling causes inner, micellar surfaces to come into play, the difference in the sizes of these accounting for the different behaviour of different adsorbents. Wool and acetate silk after swelling have larger inner surfaces than cotton. Thus an alcoholic solution of Diamine Blue 3R causes swelling of acetate silk and dyes corinth-red. After saponification with sodium hydroxide, washing the fibre, and acidifying, the colour changes to blue, showing that in saponification the inner structural conditions are changed. The reddening of blue-dyed cotton is also produced by desiccation over sulphuric acid, but to a less extent than by heating. The hypothesis of Justin-Mueller that two forms of the dye exist, one containing water and the other water-free, whilst not consistent with all experimental facts, is applicable to the portion of the dye existing in a low degree of dispersion. The observed colour changes on heating and drying may thus be connected with dehydration and simultaneous increase in the degree of dispersion. C. H. D. CLARK.

[Printing] coloured discharges on indigo grounds by means of vat dyes. C. SUNDER and R. SOLBACH (Sealed Note 2171, 22.4.12. Bull. Soc. Ind. Mulhouse, 1925, **91**, 755—757. Report by O. MICHEL, *ibid.*, 757—758).—Coloured discharges on an indigo ground are obtained by printing with a paste containing an indanthrene or helindone dye, Rongalite, anthraquinone (catalyst), and stannous hydroxide (instead of the more commonly used zinc oxide), and then passing the material, after steaming for the discharge of the indigo, through caustic soda, whereby the vat dyes are reduced and fixed. For example, for red discharges, non-mercerised indigo-dyed cotton is printed with a paste containing 70 g. of Helindone Scarlet S, 70 c.c. of glycerin of 28° B. (*d* 1·24), 160 c.c. of water, 200 g. of Rongalite C, 250 g. of a 50% solution of British gum, 40 g. of anthraquinone (paste), 30 g. of Leucotrope W, and 180 g. of 60% stannous hydroxide, then dried, steamed for 4 min., passed during 20 sec. through caustic soda of 19° B. (*d* 1·15) at 75°, washed in cold water, and soaped hot. Superior results are obtained on mercerised cotton, a paste containing 80 g. of Indanthrene Yellow G. powder, 100 c.c. of

glycerin, 150 c.c. of water, 360 g. of Rongalite C, 360 g. of a 50% solution of British gum, 60 g. of anthraquinone (paste), 120 g. of Leucotrope W, and 270 g. of 60% stannous hydroxide being used for yellow discharges and a paste containing 100 g. of Indanthrene Blue RS, 40 c.c. of glycerin, 150 c.c. of water, 200 g. of Rongalite C, 260 g. of a 50% solution of British gum, 40 g. of anthraquinone, 60 g. of Leucotrope W, and 150 g. of 60% stannous hydroxide for blue discharges. Michel draws attention to the related later processes of the Badische Anilin- & Soda-Fabrik and the investigations of Haller (B., 1923, 1127 A). A. J. Hall.

Patents.

Machines for mercerising fabrics. C. A. Gruschwitz A.-G. (E.P. 247,909, 26.6.25. Conv. 17.2.25).—The usual stenter type of machine for mercerising cotton fabric is provided with a tank through which the stretched mercerised fabric passes in a to-and-fro motion while subject to a constant weft tension and is simultaneously freed from mercerising liquor by means of water sprays. A. J. Hall.

Bleaching textiles by means of oxygen or ozone baths. R. Mohr (G.P. 421,906, 9.9.24 Addn. to 410,106 ; B., 1925, 541).—The bleaching apparatus described in the chief patent is connected through a control valve with a storage vessel to which excess of pressure liquid is supplied by a special pump. The maintenance of pressure during the process is thus assured by the pressure liquid without a separate supply of compressed air or gas. A. J. Hall.

Wetting-out means [product] for carbonisation, dyeing, etc., in which textile materials containing animal fibres are treated with a mineral acid. Chem. Fabr. Pott & Co., and F. Pospiech (E.P. 248,814, 12.11.24).—The wetting-out of textile materials with acid liquids is assisted by the presence of comparatively small quantities of the sulphonic acids of naphthalene and tetrahydronaphthalene or their alkyl derivatives together with hydrocarbons, alcohols, or ketones ; such assistants are completely stable to acids and are thus superior to those containing the salts of fatty acids. Sodium *iso*propylnaphthalenesulphonate and a mixture of sodium tetrahydronaphthalene-β-sulphonate and hexahydrophenol are suitable products. A. J. Hall.

Dyes and dyeing. [Soluble products from vat dyes.] J. Morton, J. I. M. Jones, B. Wylam, J. E. G. Harris, J. Wilson, and Morton Sundour Fabrics, Ltd. (E.P. 247,787, 11.8, 8.9, and 16.10.24, and 11.6.25).—Vat dyes in solution or suspension in a suitable organic base, are treated with an alkylsulphuric acid halide in the presence of a metal. The leuco-derivatives thus obtained are, in general, soluble in hot water and alkalis and sometimes also in acids, and may be used for dyeing animal or vegetable fibres or artificial silk. The parent dye may be regenerated on the fibre by treatment with a mild acid oxidising agent, such as acid ferric chloride. The dye may or may not be refluxed with the base and metal and cooled before the addition of the alkylsulphuric acid halide. An acid or an acid salt may be added to the mixture of dye, metal, and base. The process is applicable to indigoid dyes, such as indigo and dichlorodibromoindigo, and to anthraquinone dyes, such as benzanthrone, indanthrone, flavanthrone, pyranthrone, and anthraquinone-acridone dyes. The metal is usually zinc dust, but iron, cobalt, copper, aluminium, cadmium, tin, copper bronze, and a mixture of zinc and iron may be used. Methylsulphuric acid chloride is usually employed, but ethylsulphuric acid chloride is also suitable. Tertiary organic bases are used, usually pyridine ; dimethylaniline, quinaldine, and quinoline may be employed, and diluents, such as chlorobenzene or carbon disulphide, may be added. Suitable acidic substances are hydrochloric and acetic acids, zinc and stannous chlorides, sodium bisulphite, and pyridine hydrochloride. In general, cold water is added to the reaction mixture and the product obtained as an undissolved powder. The nature of the primary products is unknown, but there is no evidence that they are alkyl sulphuric esters ; that from indigo is a methylpyridinium derivative of indigo-sulphuric acid. Condensation is usually effected at the ordinary temperature, but with flavanthrone a temperature of about 80° is used and the metal is copper or copper bronze powder. A. Geake.

Dyes and dyeing. J. I. M. Jones, B. Wylam, J. Morton, and Morton Sundour Fabrics, Ltd. (E.P. 248,802, 25.7.24 and 25.5.25).—Stable leuco-derivatives of vat dyes capable of application to textile materials from simple aqueous solutions, the shade being developed by subsequent oxidation, *e.g.*, with ferric chloride or a dichromate, are obtained by treating the leuco-derivatives with phosphorus oxychloride or an alkylphosphoric halide in the presence of a suitable diluent such as carbon disulphide, chlorobenzene, pyridine or other tertiary amine. For example, a dark blue product slightly soluble in water and more readily soluble in dilute caustic soda and suitable for dyeing is obtained when 8 pts. of phosphorus oxychloride are added during 1½ hrs. to 3 pts. of leucoflavanthrone suspended in 8 pts. of pyridine and 25 pts. of carbon disulphide, the mixture is heated for 15 min. at 70°, then shaken with a limited quantity of water, filtered, and the residue washed free from acid. Or a reddish-violet paste containing a stable leuco-derivative of indanthrone is prepared by adding 12½ pts. of leucoindanthrone to a mixture of 30 pts. of methylphosphoric dichloride and 120 pts. of pyridine contained in a closed vessel in which air has been displaced by carbon dioxide, the mixture being maintained at 55° for 30 min. and then diluted to 2500 pts. by addition of water and filtered. A. J. Hall.

Finishing textile materials. Raduner & Co. A.-G. (G.P. 423,858, 12.5.25).—Textile materials

are impregnated with carbamide whereby they acquire a soft moist handle and are rendered partially fireproof; the carbamide may be used in conjunction with various soaps. A. J. Hall.

Treating silk. H. B. Smith (U.S.P. 1,578,933, 30.3.26. Appl., 8.3.23).—See E.P. 212,517; B., 1925, 37.

Degreasing agent [for textiles]. G. Zimmerli Chem.-Tech. Fabr. (E.P. 236,209, 20.6.25. Conv., 27.6.24).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Ammonia synthesis from coke-oven gas. G. Claude (Compt. rend., 1926, 182, 877—881).—An illustrated article in which the present position of the process is briefly considered. Hydrogen is obtained from the coke-oven gas by partial liquefaction (cf. B., 1924, 933). Nitrogen is obtained by burning some of the hydrogen in air and is then combined with hydrogen under 900 atm. in the usual Claude apparatus. S. K. Tweedy.

Neutralisation of sulphate of ammonia and notes on manufacture. C. Bateman (Gas J., 1926, 173, 748—751).—Ammonium sulphate prepared by the ordinary methods may, after "whizzing," still contain 0·3% of free acid. Washing in the centrifugal dryer with water did not leave the salt neutral, probably because part of the acidity is due to ammonium hydrogen sulphate. However, as the water did reduce the acidity, the salt was always washed with a minimum of hot water. Experiments were made with various neutralising agents and the best results were obtained with solid ammonium carbonate. After washing the ammonium sulphate in the whizzer, it was dropped into a chamber and mixed with the calculated amount of ammonium carbonate. The resulting salt contained the maximum percentage (21·10) of ammonia. On storing, it did not cake or dissociate No trouble due to pyridine was observed in the saturation process. For the removal of hydrogen sulphide from the waste gases from sulphate manufacture, oxide from the purification of oil-gas may be used. Its high tar content may be reduced by heating it by means of hot waste gases from the sulphate plant. The advantages of the semi-direct process of ammonia recovery, in which acid washers are combined with the ammonia scrubbers, are emphasised. A saving in water for removing the last traces of ammonia from the gas, less steam for the distillation of the liquor (which is less in quantity and more highly concentrated), and less effluent liquor from the still are among the economies effected. R. A. A. Taylor.

Determination of perchlorate in Chile saltpetre. G. Leimbach (Z. angew Chem., 1926, 39, 432).—The formation of the insoluble nitron salt is used as a means of determining perchlorate in Chile saltpetre. 10 g. of the sample are dissolved in a small volume of water and the greater part of the sodium is precipitated as chloride by the addition of excess of concentrated hydrochloric acid. The filtrate from the sodium chloride is concentrated: the solid which separates is dissolved in water, and nitrate and any chlorate present are decomposed by repeated evaporation with hydrochloric acid. A final evaporation to dryness suffices to drive off excess hydrochloric acid. The residual solid is dissolved in water and a little sulphuric acid added. 10 c.c. of nitron acetate (10% solution) are added to the hot solution and the mixture is slowly cooled. After keeping for 1 hr. at the ordinary temperature, the nitron perchlorate is removed, washed with a saturated aqueous solution of nitron perchlorate, and dried at 100°. The method requires modification in the presence of much potassium. L. M. Clark.

Detection and determination of small quantities of perchlorate in Chili saltpetre and in chlorates. F. L. Hahn (Z. angew Chem., 1926, 39, 451—454).—Small quantities of perchlorate in sodium nitrate solutions can be determined accurately by the colour produced by the addition of a methylene-blue solution containing a considerable quantity of zinc sulphate, the dark blue colour changing to greenish-blue and reddish-violet with increasing perchlorate concentration. By comparison with standard coloured solutions of known perchlorate content, up to 1% of perchlorate can be determined to within 0·05%. For stronger perchlorate solutions, dilution with sodium nitrate is necessary; with less than 0·1% of perchlorate, a strong methylene-blue solution is to be preferred. The process can be completed in about 1 hr., and may be applied to the determination of perchlorates in chlorate solutions. B. W. Clarke.

Detection of perborates in the presence of borates. L. Rossi (Quim. et Ind., 1926, 3, 4—5; Chem. Zentr., 1926, I., 2219).—An alkali metavanadate or pyrovanadate is reduced in aqueous solution to a vanadyl salt by treatment with sulphur dioxide, excess sulphur dioxide is expelled by boiling, and the solution is acidified with hydrochloric or sulphuric acid; on heating the solution with the borate under test, an orange-yellow coloration is produced if perborates are present, due to oxidation of the vanadyl salt to vanadate or polyvanadate. The presence of 0·01 g. of perborate in 2 g. of the borate can be detected by the reaction. L. A. Coles.

Deterioration of commercially packed chlorinated lime [bleaching powder]. C. C. McDonnell and L. Hart (U.S. Dept. Agric. Bull. 1389, Feb., 1926, 19 pp.).—The average loss of available chlorine in samples of bleaching powder, packed commercially in various containers, was 1·08% per month, over a period of 12—15 months. The rate of loss is affected by the temperature, averaging 1·44% per month during May to September and 0·61% during September to May; it is only slightly affected by the type and size of vessel in which the material is stored, deterioration being somewhat

slower when 5—10 lb. were stored in metal containers than when smaller quantities were stored in waxed fibre vessels. The rate of deterioration was lowest, however, when samples were stored in tightly stoppered, glass bottles. The visible rays of light and the presence of moisture have a slight accelerating effect upon the loss of available chlorine. Practically the whole of the loss of available chlorine is due to its conversion into "chloride" chlorine, changes in the chlorate content of the material and losses of chlorine by volatilisation being slight. L. A. COLES.

Determination of mercuric iodides. J. SANDILANDS (Pharm. J., 1926, 116, 357—358).—The mercuric iodide is dissolved in cold sodium thiosulphate solution, the mercury precipitated with hydrogen sulphide, and the mercuric sulphide filtered off, washed, dried, and weighed. For the determination of the iodine, the mercury is liberated by the addition of either zinc dust, aluminium, or magnesium and dilute sulphuric acid to a suspension of the iodide in water. The amalgam is filtered off and the iodine in the filtrate determined by Volhard's method.
E. H. SHARPLES.

Determination of carbon monoxide in hydrogen. P. SCHUFTAN (Z. angew. Chem., 1926, 39, 276—278).—When hydrogen containing carbon monoxide is led over a nickel-aluminium oxide catalyst at 200—300°, methane and water are formed according to the equation $CO+3H_2=CH_4+H_2O$. When the gas is free from oxygen and is saturated with water vapour, the carbon monoxide content may be determined by measuring the volume contraction, which is 3 times the volume of carbon monoxide. If the reaction temperature is too high or the carbon monoxide content is over 10% by volume, an error is caused by decomposition of carbon monoxide to the dioxide and carbon. For 10% of carbon monoxide the temperature should not be above 205°, but may be higher when the carbon monoxide content is lower. A. GEAKE.

See also A., April, 368, **Silicides of calcium and magnesium** (WÖHLER and SCHLIEPHAKE); **Decomposition on heating of sulphides, carbides, silicides, phosphides, silicates, and spinels with alkaline-earth oxides** (HEDVALL); **Decomposition of alkaline-earth sulphates** (ZAWIDZKI and others); **Volatility of compound of barium sulphate with sulphuric acid** (KRAUSS). 370, **Ammonium carbonates** (BONNIER); **Oxidation of combined nitrogen to potassium nitrate and reduction of potassium nitrate to cyanide** (HOFMANN and others). 371, **Bettendorf's reaction for arsenic** (ZWICKNAGL). 373, **Separation of hafnium and zirconium** (DE BOER); **Preparation of pure nickel monoxide** (LE BLANC and SACHSE). 374, **Analysis of iodides, and standardisation of permanganate and thiosulphate solutions** (ALSTERBERG). 375, **Test for iodate ion** (BICSKEI); **Reactions for nitrate and hydroxylamine** (BLOM); **Iodometric determination of arsenic acid** (ORMONT). 376, **Titration of thallous salts by potassium iodate** (BERRY); **Potentiometric evaluation of titanous chloride solutions** (KOLTHOFF, TOMICEK, and ROBINSON).

Solubility of calcium sulphate at boiler-water temperatures. HALL, ROBB, and COLEMAN.—See I.

Determination of carbon disulphide and hydrogen sulphide. HEGEL.—See V.

Recovery of tin salts from tin-plate scrap. MANTELL.—See X.

PATENTS.

Acceleration of the oxidising or condensing reactions occurring in the preparation of nitric acid from nitrous gases. N. CARO and A. R. FRANK (G.P. 424,728, 17.8.24).—When most of the nitrous gases have been condensed the cooled gases are treated with ozone, and the nitrogen pentoxide produced is converted into nitric acid.
A. B. MANNING

Apparatus for producing and recovering reaction gases [hydrogen cyanide]. M. J. BROWN, Assr. to PACIFIC R. AND H. CHEMICAL CORP. (U.S.P. 1,569,171, 12.1.26. Appl., 26.3.25).—Two liquids which produce gas on mixing, *e.g.*, acid and cyanide solution, are separately stored in reservoirs connected by conduits with a reaction chamber. The liquids are introduced through control valves into a vessel suspended from the sides of the reaction chamber and are mechanically mixed. Hydrocyanic acid gas is immediately evolved and escapes at the top of the chamber into a gas reservoir. The liquid overflows from the vessel and flows from the bottom of the chamber through an outlet chamber externally heated by steam. Further hydrocyanic acid gas is produced which passes up into the main gas exit tube. The main feature of the process is that hydrolysis is prevented by quick evolution of the gas. R. B. CLARKE

Heat interchangers for carrying out catalytic gas reactions [ammonia synthesis]. SYNTHETIC AMMONIA AND NITRATES, LTD., and F. H. BRAMWELL (E.P. 248,999, 22.7.25).—In an apparatus for the synthetic production of ammonia the cylindrical reaction chamber is surrounded by a heat exchanger in which the outgoing gases give up their heat to the ingoing gases, the whole combination being surrounded by a pressure-tight casing. To minimise the pressure loss due to friction, the gases flowing in one direction are passed in parallel through a very large number of tubes of small bore, which are made U-shaped and situated in annular passages formed by annular heat-resisting walls alternately depending and upstanding from the top and bottom of the apparatus. The limbs of each U are in adjacent annuli and all the tubes in one annulus are connected to a common header. The other stream of gases passes zig-zag up and down around the U-tubes. B. M. VENABLES.

Apparatus for the synthesis of ammonia. G. CICALI (F.P. 599,768, 17.6.25. Conv., 16.2.25).—The apparatus comprises three concentric cylinders, the innermost one being the catalyst chamber.

In the annular space between the outer and the intermediate cylinder there is a serpentine tube up which the hot reaction gases pass. The entering gases pass down the outer annular space, up through the next space containing the serpentine tube, where heat exchange takes place, and down through the last space into the catalyst chamber. By-passes permit the admixing of hot or cold gases with the mixture in the last annular space to bring it to the combining temperature. E. S. KREIS.

Fixation of solar or ultra-violet energy for producing catalytic phenomena from solar or ultra-violet rays. E. F. ROUSSEAU (E.P. 226,534, 13.12.24. Conv., 17.12.23).—Salts or solutions of salts of uranium, manganese, iron, or nickel are subjected to the action of solar or ultra-violet radiation, and then added to liquids in which photocatalysis or the sterilisation or activation of ferments is to be effected. J. S. G. THOMAS.

Manufacturing colloidal compounds of arsenic. A. CHWALA, Assr. to ODERBERGER CHEM. WERKE A.-G. (U.S.P. 1,573,375, 16.2.26. Appl., 20.8.24).—The arsenic compound is dispersed in the presence of protective colloids, and the salts of metallic acids, *e.g.*, calcium plumbate, sodium antimonate, with or without the addition of compounds of high molecular weight forming compounds of a complex nature with the insoluble arsenic compound, *e.g.*, protalbinic acid. E. S. KREIS.

Double salts of thiocyanic acid. H. FRIEDENTHAL (F.P. 598,766, 25.5.25).—A solution of a thiocyanate and the bromide or iodide of a volatile base, such as ammonia or hydrazine, is added to an alkaline-earth salt of a volatile weak acid, and the ammonium or hydrazine salt of the volatile acid which is formed is driven off. The double salts produced find application for photographic, disinfectant, and other purposes. A. B. MANNING.

Preparation of cyanides from cyanamide salts. N. CARO and A. R. FRANK (Austr. P. 101,316, 14.8.24).—A powdered mixture of a cyanamide salt and carbon, *e.g.*, commercial calcium cyanamide, together with a flux such as sodium chloride, falls through an arc directly on to a cooled surface under such conditions that the molten material solidifies immediately in as thin layers as possible.
A. B. MANNING.

Manufacture of pure alumina. J. D. RIEDEL A.-G. (G.P. 424,701, 31.12.19).—A solution of alumina containing iron is obtained by decomposing iron-bearing aluminosilicates with ammonium sulphate and lixiviating. From this solution, either directly or after removal of the iron, by addition of ammonium sulphate and ammonia, solid aluminium sulphate or insoluble basic sulphate is prepared. These compounds are converted by the action of ammonium sulphate and ammonia or of ammonia into the solid hydroxide. The pure hydroxide, then obtained directly by the action of steam, or, if iron is present, after a previous purification by the Bayer process, is finally calcined. A. COUSEN.

[Manufacture of] sodium peroxide. VEREIN FÜR CHEM. U. MET. PRODUKTION (Swiss P. 112,963, 16.10.24).—In an apparatus for producing sodium peroxide by burning sodium in one chamber and oxidising the sodium oxide so produced in a second chamber, the sodium oxide is obtained as a porous mass so that it can be easily oxidised, by preventing undue rise of temperature in the first chamber by regulating the amount of air admitted.
E. S. KREIS.

[Manufacture of] sulphuryl chloride. R. M. MCKEE and C. M. SALLS (Can. P. 251,586, 25.6.24; cf. B., 1924, 378).—Chlorine and sulphur dioxide are passed into a mixture of sulphuryl chloride and an inert diluent containing active charcoal in suspension. The mixture is cooled to prevent rise in temperature. E. S. KREIS.

Liquefaction and rectification of air or other gaseous mixture into its components. G. CICALI (E.P. 248,977, 16.6.25).—The purified air is compressed to a few atmospheres in a low-pressure cylinder surrounded by cooling water. One portion of the issuing air passes to a heat exchanger and is cooled by cold, rectified nitrogen passing in the opposite direction. Thence it passes into the bottom of the rectifying column. The other portion is conveyed to water-cooled compressors and compressed to the maximum pressure used; it then enters the intermediate tube of a three-pipe heat exchanger, the outer tube of which contains cold, rectified nitrogen, and the inner tube, cold, rectified oxygen. The air issuing from this exchanger is subdivided. One part is expanded (and employed to do work) and then introduced into the foot of the column; the other part is liquefied, without altering its pressure, in a coil surrounded by liquid nitrogen. The air passing up the column meets a baffling device which causes oxygen to condense at the foot.
R. B. CLARKE.

Continuous separation of sulphur from sulphur solutions. E. LEGELER (E.P. 249,044, 26.11.25. Conv., 6.7.25).—The sulphur solution is introduced at the top of a heated cylindrical vessel, and drips on to a heating device situated about half-way up the vessel. The device is in the form of a segment of a sphere, or conical, and is heated to the boiling point of the solvent and at least to the melting point of sulphur. Sulphur melts on the plate, falls to the bottom of the vessel, and is siphoned off. It is entirely free from solvent. The rising solvent vapours are withdrawn and condensed.
R. B. CLARKE.

Producing purified sulphur. C. MARX, Assr. to UNION SULPHUR Co. (U.S.P. 1,574,989, 2.3.26. Appl., 19.12.22; cf. following abstract).—Sulphide ores, which may also contain free sulphur, are distilled under a high vacuum and the sulphur vapours are condensed in a vacuum receiver. In the case of iron pyrites the reaction, $FeS_2 = FeS + S$, takes place, and any free sulphur originally present is also isolated. R. B. CLARKE.

Producing purified sulphur from impure sulphur or ores containing it in elemental form. C. Marx, Assr. to Union Sulphur Co. (U.S.P. 1,574,988, 2.3.26. Appl., 19.12 22; cf. preceding abstract).—The sulphur-containing material is distilled under a high vacuum, and the sulphur vapours are condensed in a vacuum receiver The physical nature of the sulphur may be modified by varying the conditions of temperature and pressure; thus it may be obtained either molten or as a fine granular powder. R. B. Clarke.

Separation of sulphur from gases containing hydrogen sulphide, and free from ammonia. F. Muhlert (G.P. 423,395, 18.3.24).—The gases are washed with a solution containing a mixture of cupric chloride with ammonium chloride, or an alkali, alkaline-earth, or earth metal chloride, in the proportion of at least 1 mol. of the former to two of the latter. To separate the sulphur from the copper sulphide sludge so obtained, the latter is heated with an amount of the original cupric solution (or similar copper solution) at least equivalent to the cupric sulphide. The solution then remaining, which contains a double salt of cuprous chloride, is oxidised by air, oxygen, or chlorine, and the cupric chloride olution thereby regenerated. A. B. Manning.

Extraction of hydrogen from gaseous mixtures. Soc. d'Etudes et de Constructions Métallurgiques (F.P. 599,895, 27.9.24).—The gases containing the hydrogen are passed along one side of a permeable partition under a slight pressure, whilst an indifferent gas, such as sulphur dioxide or ammonia, which is easily separated from the hydrogen, is led along the other side, under a slightly reduced pressure. E. S. Kreis.

Manufacture of hydrogen by partial liquefaction of gaseous mixtures. G. Claude, Assr. to Lazote, Inc. (U.S.P. 1,579,348, 6.4.26. Appl., 7.1.22).—See E.P. 174,327; B., 1922, 463 A.

Recovering soda from solutions (E.P. 248,864).—See V.

VIII.—GLASS; CERAMICS.

Preparation and properties of light-dispersing glasses. G. Schott (Glastech. Ber., 1925, 3, 315—329).—Two chief types of such glasses may be distinguished, (1) transparent glasses with specially shaped or treated surface, (2) glasses inhomogeneous throughout. In the first class are "Holophane" (glass with parallel ribbing on the inner surface), sand-blasted, and etched ware. The matt etch of hydrofluoric acid varies according to the method of procedure and is, in general, less coarse than the sand etch. In the second class are alabaster, opal, and milk glasses. Alabaster glass is formed by devitrification, usually in a potash-lime-silicate of low lime content. Stannic oxide also gives a crystalline deposit on cooling the glass in which it is dissolved, and recently zirconia, titania, and sodium chloride have similarly been used for producing opal glass. Opal effects are obtained by using fluorine-containing materials or by phosphates which form emulsions on cooling. The composition of the disperse phase has not been definitely determined, but aluminium fluoride or sodium silicofluoride has been suggested. Wo. Ostwald gives three causes for light scattering, viz., refraction, reflection, and diffraction, usually two or more being operative at the same time. In the first two cases determinative factors are the refractive index of the glass, the difference of refractive index of the two phases, and the size and number of the particles. Dispersion of light by refraction and reflection results in chromatic resolution, but with a large difference of refractive index of the two phases the colour phenomenon is masked. When the size of the particles is of the order of a wave-length of visible light, diffraction becomes the most important factor, the scattering thereby increasing with the degree of division until the particle size becomes less than that of a light wave-length, when scattering once more decreases. Colour effect due to opalescence is more important than that due to absorption, and it gives usually violet-blue reflection and yellowish-red transmission. Opalescent colours are most marked with a highly disperse phase, though colour effects may occur through refraction when large particles are present. The author has prepared a phosphate glass which in thin layers allows light to pass unchanged in colour. The glass contains relatively few particles which, in consequence of their large size, give good scattering and little absorption. Absorption and reflection losses are small, the following being percentage light loss on transmission through various glasses:—clear glass 5·5, etched glass 7·5, sand-blasted glass 11·2, casing glass (*a*) 9·3 (thin), (*b*) 11·5 (thick), (*c*) 27·0, Jena milk glass (massive) 19·5, Peters glass 25·2. The light-distribution curves of six glasses are given. The efficiency of a light-dispersing glass may be numerically represented by the formula E = scattering power/absorption. A. Cousen.

Nomenclature and classification of optical glasses. P. Nicolardot (Rev. gén. Colloïd., 1926, 4, 9—13, 39—50).—Methods of classification of optical glasses suggested by Zschimmer, Zschokke, Morey, Peddle, and Montgomery were examined. The schematic diagram of Montgomery (B., 1920, 572) is modified, as a basis of classification, by plotting the logarithm of dispersive power against the index of refraction. Since the region richest in glasses is then cramped a division into three diagrams is proposed, corresponding to visual ($x = n_D$, $y = \log V_D$, $V_D = [n_F - n_C]/[n_D - 1]$), photovisual ($x = n_F$, $V_F = [n_G - n_D]/[n_F - 1]$), and photographic ($x = n_G$, $V_G = [n_H - n_F]/[n_G - 1]$). The method of classifying recently adopted by the firm of Schott is commended. A. Cousen.

Patents.

Apparatus and process for making glass. R. R. Shively, Assr. to B. F. Drakenfeld & Co. (U.S.P. 1,575,514, 2.3.26. Appl., 12.4.24).—The batch is placed within the furnace chamber so as to cover only a minor portion of the floor thereof, at a

point near one end. It is heated uniformly over the surface by suitable arrangement of the heating medium, and fusion takes place with a progressive and uniform reduction of the bulk. As the glass is formed it is led away to an outlet at the opposite end of the chamber. A. COUSEN.

Glass batch. CORNING GLASS WORKS, Assees. of W. C. TAYLOR (Can. P. 249,088, 26.7.24).—Essentially, the material is free from silica, and contains up to 65% of boric oxide, at least 15% of alumina, up to 10% of potassium oxide, and the oxide of an element of the second group of the periodic system. A. COUSEN.

Furnaces. R. MALVOS and M. CROZEMARIE (E.P. 248,036, 19.11.24).—A furnace in which a temperature of 1800° is obtainable from solid fuel, such as hard coal, and which is suitable for burning refractory products such as abrasives, comprises three or more intermittent burning chambers in conjunction with a common regenerator. The burning chambers are charged with the goods through holes in the roof which are then sealed up and the goods not touched till cool again at the end of the burning. Each chamber has several U-shaped furnaces, each with two grates and water troughs forming ash-pits. The furnaces are first fired slowly using cold air, but later hot air from the regenerator is used. After the burning the charge in any one chamber is progressively cooled by drawing out hot gases from the chamber with admission of an equivalent quantity of cold air. B. M. VENABLES.

Manufacture of refractory material. L. LONGCHAMBON and A. TRAVERS (F.P. 599,517, 13.6.25).—The material is obtained by heating to 900—1470° quartzite rock in the presence of 0·5—5% of an alkali salt, whereby the quartz is converted into tridymite or cristobalite, or into a mixture of these. L. A. COLES.

Enamelling metal. W. J. BECK and J. A. AUPPERLE, Assrs. to AMER. ROLLING MILL Co. (U.S.P. 1,578,706, 30.3.26. Appl., 16.11.23).—See E.P. 239,902 ; B., 1925, 962.

Annealing furnaces [lehrs]. C. E. FRAZIER (E.P. 249,636, 9.2.25).

IX.—BUILDING MATERIALS.

Aluminous cements. H. EISENBECK (Chem.-Ztg., 1926, **50**, 165—167, 202—204, 239—240, 245—248).—The chemical properties and constituents, and therefore to a large extent the value as a building material of aluminous cements, are represented by their position in the lime-silica-alumina system plotted on the Gibbs triangular co-ordinate method. Aluminous cements develop a strength in 24 hrs. equal to that of Portland cement in 28 days ; this rapid hardening is accompanied by a slow setting, which is of great importance in building operations. The hardening process is accompanied by a considerable rise in temperature which enables the cement to be used in cold weather with the minimum of protection. Aluminous cement offers much greater resistance than Portland cement to attack by waters containing alkalis, acids, sulphates, or magnesium salts and is therefore very valuable in sea water work. The price of the cement is double that of Portland cement, but a much leaner mix can be used and the rapid hardening properties permit of economy in time and in initial outlay on forms, shuttering and the like. B. W. CLARKE.

Producing a hydraulic cement from copper slag. G. AGDE and P. ASSMANN (Z. angew. Chem., 1926, 39, 271—276).—Slag remaining from the recovery of copper from its alloys with base metals, and containing 42% SiO_2, 13·2% FeO, 1·7% Fe_2O_3, and 23·5% CaO in addition to copper, aluminium, zinc, and magnesium, can be used for the production of a valuable cement by an ordinary oxidising burning with the addition of lime ; the most suitable proportion must be determined empirically. The presence of cupric and cuprous oxides is not detrimental. Phosphoric acid greatly diminishes the strength if more than 0·6% P_2O_5 is present in the finished cement. If 3% of gypsum is added setting begins more slowly but complete setting is accelerated. A. GEAKE.

Rapid determination of silica and lime in the raw mixture for blast-furnace cement. F. STRUMPF (Z. angew. Chem., 1926, 39, 278—279).—Silica is determined by evaporating the powder with concentrated hydrochloric acid, treating the residue with dilute acid, filtering, and weighing. The filtrate is made alkaline with ammonia and then acid with acetic acid. Iron and aluminium are precipitated as phosphates and, without filtering, calcium is precipitated as oxalate. The combined precipitates are purified by redissolving and precipitating, and are finally dissolved in sulphuric acid and calcium determined by titrating the oxalic acid with permanganate. A. GEAKE.

Curing concrete in a semi-arid climate. C. L. MCKESSON (Eng. News-Rec., 1926, **96**, 452—453 ; cf. Gonnerman and McKesson, B., 1926, 14).—Further tests on concrete pavement laid down during the dry season in California, with a day humidity of 15—40%, show that watering for seven days after laying is sufficient to give the concrete maximum strength at 90 days ; in an emergency, watering for three days would give satisfactory results, both from the points of view of strength and of surface hardness. Protecting the surface from evaporation by dry earth gives a concrete which does not increase much in strength after about 14 days, as the original water used for mixing is not sufficient for the final hydration process on which the ultimate strength depends. Covering the surface with flake calcium chloride gives fairly good results and its use is justified where water is not available. B. W. CLARKE.

Wood impregnation by the Cobra process. R. NOWOTNY (Z. angew. Chem., 1926, 39, 428—431).—Inspection of cross-sections of spruce poles, which have been treated previously for immunity from rotting

and wood diseases by injecting an aqueous solution of sodium fluoride, sodium dinitrophenoxide and a little zinc chloride, shows that after several months, complete diffusion of sodium fluoride across the section has occurred. Sodium dinitrophenoxide diffuses more slowly, but after some months, a section shows that a complete annulus of this reagent has spread around the circumference of the pole, and that in general, the depth of penetration is determined by the depth of the injection puncture. In both cases, diffusion takes place more readily in the direction of the "annual" rings than radially. The Cobra method of wood preservation (injection of the preservative solution through boreholes drilled to a depth of several cm. in the neighbourhood of the danger zone) offers advantages over older processes, since it is not necessary to use seasoned poles, as the presence of moisture in the wood renders diffusion more rapid, while the necessity for storing the timber during seasoning is consequently avoided. The danger that water-soluble impregnating materials may be slowly dissolved out of the timber by long contact with moist soil, is negatived by the fact that a pine pole impregnated sixteen years previously with sodium fluoride still had this salt diffused throughout its cross-section. L. M. CLARK.

PATENTS.

Kiln for burning cement and for similar purposes. C. NASKE (E.P. 227,444, 6.1.25. Conv., 8.1.24).—An automatic shaft furnace is constructed so that the raw material together with the fuel is blown in from underneath, and the completely burnt material, owing to its increased specific gravity, falls and leaves the furnace at the inlet for the mixture, while the incompletely burnt material leaves at the top of the furnace together with the combustion gases and is collected in a dust chamber, and returned to the furnace to complete the burning process. B. W. CLARKE.

Rotary kilns for burning cement, ore and similar materials. VICKERS, LTD., and L. D. PARKER (E.P. 248,079, 27.11.24).—In a rotary kiln, a heat-recuperating device is attached to the lower or clinker outlet end of the main rotating cylinder, and is so constructed that the clinker moves in circuitous passages counter-current to the incoming air for combustion. B. M. VENABLES.

Manufacture of aluminous cements. E. MARTIN (F.P. 597,978, 30.4.25).—To the initial materials small amounts of alkali salts (as chlorides, sulphates, carbonates, silicates, or aluminates), calcium chloride, calcium fluoride, or cryolite are added, and the mixture is heated to fusion, sintering, or to a temperature at which neither fusion nor sintering occurs. The addition facilitates the formation of hydraulic products which set more rapidly than the untreated material. A. COUSEN.

Manufacture of cement. H. LOESCHER (F.P. 599,104, 4.6.25. Conv., 15.10.24, and 599,507, 12.6.25). —Clinker rich in lime is quenched with water, the powdery portion is separated, and the lumpy portion ground and again mixed with the powder. Blast-furnace slag is added, and the mixture dried, or completely slaked as desired. Alternatively, clinker rich in lime or hydraulic lime may be slaked by means of moist, granulated, blast-furnace slag. A. COUSEN.

Manufacture of cement. SOC. D'EXPLOIT. DES PROC. IND. CANALOT (F.P. 599,286, 9.6.25).—Cement is manufactured in vertical kilns, a portion of the raw material being moistened with a paste made by mixing raw material with water. L. A. COLES.

Cooling and hydrating cement clinker from rotary kilns. F. KRUPP GRUSONWERK A.-G., Assees. of C. MITTAG (G.P. 424,065, 20.3.25. Addn. to 410,325).—Additional steam for hydrating the clinker in the process described in the chief patent (cf. B., 1925, 633), is generated in the cooling chamber and is added therein to the mixture of air and steam. The waste heat of the gases withdrawn from the apparatus, is utilised in heat-exchangers. L. A. COLES.

Manufacture of lime-silica bricks. A. A. COUTANT (F.P. 599,918, 22.4.25).—The mixture of lime, silica, and water used for the production of the bricks is treated with steam in a rotating autoclave in order to ensure complete slaking of the lime. L. A. COLES.

Bituminous emulsions. G. S. HAY (E.P. 248,859, 16.12.24).—The stability of an aqueous bituminous emulsion is improved by mixing therewith, after manufacture, up to 10% of a stabilising agent comprising one or more compounds soluble in or miscible with water and which have the property of lowering the freezing point of the emulsion without causing disruption. Suitable compounds are monohydric, dihydric, or polyhydric alcohols of the paraffin, olefine, or acetylene series, *cyclo*hexanol or hydrogenated phenols; glucose and other sugars; dextrin and soluble carbohyrates. B. W. CLARKE.

Laying the dust on roads. ALLCHEMIN ALLGEM. CHEM. IND. A.-G., and R. LICHTENSTERN (F.P. 599,497, 12.6.25. Conv., 7.8.24).—Sodium or potassium naphthenate is added to oil for spraying on roads to lay the dust. Calcium naphthenate is formed by interaction with lime or calcium carbonate in the surface material, and prevents subsequent oxidation of the oil. L. A. COLES.

Product for impregnation of wood. P. R. CHAMBIGE (E.P. 249,698, 25.5.25).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Effect of the physical properties of ore and coke on the capacity of the blast furnace. T. L. JOSEPH, P. H. ROYSTER, and S. P. KINNEY (Proc. Eng. Soc. W. Pa., 1926, 41, 428—459).—The relations existing between the physical properties of coke and ore and the capacity of the blast-furnace as investigated

previously by the U.S. Bureau of Mines (cf. Sherman and Blizard, B., 1923, 436 A; Royster and Joseph, 1924, 470) are correlated with blast-furnace practice. Variation in the combustibility of coke or in the quantity of air blown did not alter the shape of the combustion zone, which, however, was shortened by decrease in size of the coke. The capacity of the furnace is believed to depend on the rate of gasification of carbon at the tuyère, but this rate is limited by the speed of reduction in the upper part of the shaft, which itself is dependent on the degree of contact between the ore particles and the reducing gas. C. A. KING.

Diminution and behaviour of the sulphur contained in producer-gas in the Siemens–Martin furnace. J. BRONN (Stahl u. Eisen, 1926, **46**, 78—80).—Experiments were made by adding 2·3% of burnt lime to the coal containing 1·4% S to be gasified in a Morgan gas producer, and the effect on the sulphur content of the slag formed without fluorspar additions in a Siemens–Martin furnace was determined. After addition of the lime 2—4% of metallic iron was found in the producer slag or ash, the iron being the principal carrier of the sulphur. The sulphur content was increased from 1·54% to 2·79% and diminished in the Siemens–Martin slag from 0·27% to 0·188%. The gas producer slag per 100 kg. of coal gasified had retained more than twice the amount of sulphur, and the sulphur in the Siemens–Martin slag was diminished by roughly a third. The sulphur in the bath remained approximately the same, so that the amount taken up thereby from the gases was negligible. A lime addition of 3·5% to the coal produced no further decrease in the sulphur content of the Siemens–Martin slag and freshly slaked lime was not so effective as burnt lime. The Siemens–Martin slag does not take up sulphur as sulphur dioxide but in the form of sulphur compounds not containing oxygen for which it has a much greater affinity. The lime additions in the producer diminished the tendency of the coal to clinker, and the slag was more friable. Even with lignite it is advantageous to add lime. T. H. BURNHAM.

Pre-melting pig-iron for use in the scrap-pig iron process in Siemens-Martin furnaces. E. HERZOG (Stahl u. Eisen, 1926, **46**, 357—361).—Consideration of published data (cf. Ber. Stahlw.-Aussch. V. d. Eisenh., 1925, [98], and Iron Age, 1921, **108**, 403—405), indicates that the use of cupolas for premelting pig-iron for the manufacture of steel by the Siemens-Martin process is more economical than direct melting of pig-iron in the Siemens-Martin furnace and slightly more economical than the use of molten pig-iron direct from a blast furnace. The economy is ascribed chiefly to two factors, (*a*) a shortening in the time required for melting, (*b*) a decreased loss of iron in the slag. Moreover, since most of the silicon is removed as calcium silicate during the premelting process, pig-irons of high silicon content, which otherwise could not be used in a Siemens–Martin furnace, are made available. Premelting cannot be used economically to obtain steels of less than 0·04% sulphur content. L. M. CLARK.

Smelting experiments with Fricktaler and Gonzen iron ores. R. DURRER (Stahl u. Eisen, 1926, **46**, 328—332).—The experiments were carried out at the Oehler Iron and Steel Works in a one-phase electric furnace with a capacity of 450 kw. The furnace was lined with waste material from the electrodes. The charge was broken up into pieces the size of the fist. Both coke (small) and charcoal were used as reducing materials with spathic iron ore as flux. A record of 20 tappings is given, also analyses of the materials used and the iron and slags produced. In the earlier batches, using Fricktaler ore, and coke, the iron produced had a normal carbon content, the phosphorus passed almost entirely into the iron, and desulphurisation was considerable, the slag containing less sulphur than that introduced by the flux. When charcoal was used the iron showed a lower carbon and sulphur content but was high in silicon (up to 11·5%). On charging with Gonzen ore, which is more difficult to reduce, the silicon content of the iron fell to 3—6%, the phosphorus decreased, and the sulphur increased in accordance with the composition of the ore. Analysis of the slags showed less than 0·7% of carbon when coke was used but 2·2 to 15% with charcoal as reducing material. This is attributed to the higher temperature and consequent formation of calcium carbide. In large-scale practice, the higher temperatures which occur bring about the same effects when using coke as only happen in small furnaces when charcoal is used. A. COULTHARD.

Nitridation of ordinary and special steels. L. GUILLET (Compt. rend., 1926, **182**, 903—907).—A new method of case-hardening steel, which is being applied technically, consists in heating the material at 500—510° for 6 hrs. in a current of ammonia. Investigations conducted with a number of special steels show that an increase in surface hardness occurs with alloys containing chromium, silicon, molybdenum, and aluminium. The increase in hardness is particularly marked in the case of a steel with a high content of aluminium, and this promises to have technical developments. The surface-hardening effect is attributed to the influence of the added elements in decreasing the diffusion of nitrogen by the formation of nitrides. C. J. STILL.

Utilisation of chlorine in recovery of tin and tin salts from tin-plate scrap. C. L. MANTELL (Trans. Amer. Electrochem. Soc., 1926, **49**, 87—95. Advance copy).—A historical survey of the development of the chlorine process for detinning tin-plate scrap is given together with a brief discussion of its economics and commercial adaptation. Essentials for successful work are perfectly dry and clean scrap free from grease, varnish, and other organic matter, efficient cooling to keep the temperature in the reaction vessel below 38°, and thorough washing of the residual iron to remove traces of ferric chloride. The cost of the process is almost completely covered by the sale of the detinned scrap, so that the value of

the tin or stannic chloride recovered is almost entirely profit. A. R. Powell.

Examination of badly corroded brass condenser tubes. M. von Schwarz (Korrosion u. Metallschutz, 1926, 2, 9—17; Chem. Zentr., 1926, I, 2406).—After less than 1 year's use brass condenser tubes from an electricity generating station were badly corroded; the insides of the tubes were covered irregularly with numerous bright patches arranged along the lines caused by drawing and extensive dezincification had taken place. The deposits contained 54·9% ZnO, 21·6% CO_2, 7·9% SiO_2, and 6·5% H_2O, whilst slightly corroded portions of the tube assayed 60·46—62·04% Cu, 36·20—38·83% Zn, 0·33—0·78% Pb, 0·14—0·93% Fe, and a trace of arsenic. The cause of the corrosion appeared to be the use of water containing sodium hydrogen carbonate formed during softening by the permutit process; this decomposed on heating giving the normal carbonate which attacked the β solid solution, without seriously affecting the α constituent. It is recommended that brass condenser tubes should consist entirely of α, should contain a small proportion of arsenic, and should be annealed to obtain a coarsely crystalline structure. A. R. Powell.

Phosphor-bronze. Thermal analysis of the system copper-phosphorus-tin. L. C. Glaser and H. J. Seemann (Z. techn. Physik, 1926, 7, 42—46; Chem. Zentr., 1926, I, 2049).—The part of the system Cu-Sn-P which is of technical importance, namely, the corner in the neighbourhood of copper, has been investigated thermally and microscopically up to a content of 25% Sn and 8% P. The existence of a ternary eutectic of m.p. 628° at 80·7% Cu, 4·5% P, 14·8% Sn, has been established. The exact position of the saturation limits of the Cu-mixed crystals, could not be determined by this method, owing to the marked liquation phenomena.

A. B. Manning.

Determination of chromium in chrome iron ore. E. Dittler (Z. angew. Chem., 1926, 39, 279).—Chromium has been determined in 19 samples of chromite from Uskub by the iodometric and permanganate methods. Of the three empirical factors, 0·310, 0·3109, and 0·3165 (theoretical 0·3105), recommended for the latter method, 0·3109 gives results agreeing best with the iodometric method.

A. Geake.

See also A., April, 364, **Electrochemical precipitation of copper from its salts by zinc** (Galecki and Orlowski; Galecki and Kuczynski). 374, **Determinations of carbon in steel based on measurement of gaseous volume** (Hackspill and d'Huart). 376, **Precipitation of magnesium with aluminium hydroxide** (Lassieur); **Time factor in de Haen-Low method of determining traces of copper** (Dunncliff and Ram). 377, **Detection of titanium and uranium** (Tananaev and Pantschenko); **Separation of germanium** (Wada and Kato); **Determination of iron by dichromate method** (Besombe); **Determination of gold dissolved in mercury** (Tammann and Kollmann).

Cement from copper slag. Agde and Assmann.—See IX.

Tin-plate for containers for food products. Serger.—See XIX.

Patents.

Open hearth furnaces. E. Bosshardt (E.P. 247,509, 9.7.25. Conv., 10.2.25. Addn. to 223,870).—In mild steel produced by furnaces of the type described in the main patent (B., 1925, 456), blowholes and blisters are caused by the fact that gas from the generators is forced under pressure into the melting chamber, rendering it difficult to free the molten metal from gas. To overcome this difficulty the furnace is modified so that gas is drawn into the melting chamber by the suction of the furnace chimney, causing a partial vacuum about the molten charge. The furnace heads and gas generators are air-tight, and an adjustable inlet mechanism is provided below the grate to admit the air necessary for combustion of the fuel. Steel with a carbon content of 0·05%, and practically free from oxygen, has been produced during continuous working, while fuel consumption is reduced by 40%. L. M. Clark.

Treating silicon steel sheets. Brit. Thomson-Houston Co., Ltd. From Gen. Electric Co. (E.P. 248,140, 6.1.25).—Rolled steel sheets, with a silicon content of 3—5%, for use in transformer cores, have their magnetic permeability increased by about 15% at the working density of magnetic flux and the hysteresis watt loss decreased by 20% when they are annealed at 800—850° under conditions favourable to the removal of deleterious gases, followed by treatment with a pickling solution of dilute acid, which removes a superficial surface layer of the sheet in addition to the scale. Somewhat less effective is a preliminary pickling treatment with subsequent annealing in hydrogen. L. M. Clark.

Apparatus for reducing iron ore and iron cinders from pyrites to metal and for other purposes. F. L. Duffield (E.P. 248,418, 3.10.24).—Iron ore or pyrites cinder is introduced into and passes through a rotary furnace having the shape of a truncated cone. From this furnace the calcined material is conveyed by means of a shoot into a second similar furnace situated at a lower level in which the ore is reduced. Means are provided for introducing air and fuel into the respective furnaces, which are both mounted so that they can be tilted.

C. A. King.

Heat treatment of steels and other metals. F. B. Dehn. From E. G. Budd Manuf. Co. (E.P. 248,801, 15.9.24).—Excessive grain growth in low-carbon steels and metals of the so-called ductile metal class, occasioned by annealing the metals after cold working, is prevented by maintaining the cold-worked metal for a suitable period at a temperature below that at which grain growth develops and then raising the temperature to the normal annealing point. C. A. King.

Heat treated alloy steels containing zirconium. H. E. Potts. From Electro Metallurgical Co.

(E.P. 248,996, 14.7.25).—The properties of steels containing alloying elements of the class which form structurally free carbides in the fully annealed alloy steels, but other than or in addition to chromium, *e.g.*, manganese, molybdenum, tungsten, vanadium, are enhanced if zirconium (*e.g.* 0·03—0·5%) is incorporated in the alloy, particularly when the Brinell hardness is greater than 300. C. A. KING.

Metal chip briquette. G. E. GAIL, JUN. (U.S.P. 1,574,878, 2.3.26. Appl., 2.2.25).—A briquette adapted for use in cupolas is composed of a dried compacted block of metallic chips with a binder consisting solely of diluted water-glass. The briquette withstands high temperatures without disintegration. L. M. CLARK.

Beneficiation of [iron] ores. C. P. MCCORMACK (U.S.P. 1,575,852, 9.3.26. Appl., 5.5.25).—Oxidised iron ores containing aluminium are roasted with sodium carbonate in order to convert a definite amount of the alumina into soluble sodium aluminate which is then removed by leaching. A. R. POWELL.

Direct production of steel or malleable iron from ore. F. BURGERS and K. KINTZINGER (G.P. 424,184, 29.10.21).—Formation of slag and loss of iron by oxidation in the direct production of steel from ore are reduced to a minimum by passing the reducing gases through a shaft connected with the hearth, and packed with an agglomerated mixture of ore and flux. L. A. COLES.

Decarbonisation of ferrochromium. SIEMENS & HALSKE A.-G., L. FREDERICH, and W. RODENHAUSER (F.P. 600,079, 25.6.25. Conv., 26.6.24).—Ferrochromium is decarbonised by treatment while heated to 1400—1475° in an induction furnace, with air, air enriched with oxygen, steam, or peroxides. Absorbed nitrogen is removed by treatment with, *e.g.*, titanium, calcium, or aluminium. L. A. COLES.

Alloys and alloy articles. WESTERN ELECTRIC CO., INC. From WESTERN ELECTRIC CO., LTD. (E.P. 248,458, 3.12.24).—Alloys are prepared in such a manner that a supersaturated solid solution is produced. For example a lead alloy containing 2·5% Sb is heated at a temperature below the eutectic fusing temperature, quenched, and aged at atmospheric temperature. Articles of such an alloy may be cast, rolled, or forged and afterwards quenched and aged. C. A. KING.

Treating [pickling] metal. W. C. KRONQUEST, Assr. to ACME STEEL CO. (U.S.P. 1,574,823, 2.3.26. Appl., 27.5.25).—Metal is pickled by the electrolysis of a solution of acid using the metal as anode and a material not affected by the acid as cathode. Adsorption of hydrogen by the metal is thereby diminished. C. A. KING.

Refining gallium metal. S. BOYER. Assr. to GEN. ELECTRIC CO. (U.S.P. 1,576,083, 9.3.26. Appl., 12.8.25).—A film of halogen compound is formed on the surface of molten gallium and the temperature is raised in a vacuum until this film is completely volatilised. A. R. POWELL.

Forming a protective coating on metals. METALLBANK U. METALLURGISCHE GES. A.-G. (F.P. 599,974, 22.6.25. Conv., 9.1.25).—Metals such as aluminium, magnesium, or calcium are coated by electrolytic, chemical, or mechanical means with a protective layer consisting of cadmium (preferably), selenium, lead, chromium, tungsten, molybdenum, manganese, cobalt, bismuth, zinc, antimony, or tin, or alloys of these metals. L. A. COLES.

Recovery of zinc from fine ores or other material containing it. METALLBANK U. METALLURGISCHE GES. A.-G. (G.P. 421,384, 11.5.16).—The material, *e.g.*, burnt pyrites containing zinc, is sintered and desulphurised, *e.g.*, in a Dwight–Lloyd furnace, and the zinc is volatilised and recovered by heating the product in a shaft furnace. L. A. COLES.

Welding electrodes. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of R. T. GILLETTE (E.P. 237,902, 23.7.25. Conv., 30.7.24).—See U.S.P. 1,539,810; B., 1925, 598.

Purified sulphur from sulphide ores (U.S.P. 1,574,987).—See VII.

Rotary kilns (E.P. 248,079).—See IX.

XI.—ELECTROTECHNICS.

Recent improvements in the lead accumulator. C. FÉRY (Bull. Soc. d'Encour., 1926, 125, 49—60; cf. B., 1925, 997).—It is shown that local action and the secondary reaction previously proposed quantitatively account for the discrepancies between the results of Chèneveau's experiments and the results predicted by the author's theory of accumulator discharge. The sulphation accompanying the storage of discharged cells is attributed to the oxidation of the plumbous sulphate formed in the negative grids during discharge: $Pb_2SO_4+O+H_2SO_4=2PbSO_4+H_2O$. An accumulator which will not sulphate is described; the negative grid is placed horizontally underneath the positive so that it is not accessible to atmospheric oxygen or to the oxygen which may be produced at the positive plate by spontaneous decomposition of the Pb_2O_5. S. K. TWEEDY.

See also A., April, 362, **Electrochemical behaviour of chromium** (GRUBE, HEIDINGER, and SCHLECHT). 365, **Water and metals under the influence of electrolysis** (SAXON). 366, **Electrolysis of aqueous solutions of hydrogen chloride** (HOLLARD); **Oxidation of manganous ion to permanganate** (CAMPBELL). 374, **Errors in electro-analysis** (BOEHM). 376, **Use of water-alcohol mixtures in electrometric study of precipitation reactions** (ATHANASIU). 378, **Simple hydrogen electrode** (SIDERIS).

PATENTS.

Electrodes for the purification of gases. SIEMENS-SCHUCKERTWERKE GES. M. B. H. (E.P.

235,582, 11.6.25. Conv., 12.6.24).—Precipitation electrodes which resist shock and corrosion and which may be sawn and nailed are made from fibrous asbestos materials, slag wool, hair, or wood, with a semi-conducting binder, such as cement, plaster, magnesium oxide or chloride. Graphite or oxides, or other conducting compounds of metals may be added to increase the conductivity. J. GRANT.

Electrical precipitation of suspended particles from gases. SIEMENS-SCHUCKERTWERKE G.M.B.H. (Swiss P. 112,962, 14.11.24. Conv., 30.11.23).—The potential and the gas velocity are so regulated that the particles are not precipitated on the electrodes, but pass away with the gases and are subsequently removed through baffled openings in the channel walls. E. S. KREIS.

Electric incandescence lamps. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of G. R. FONDA (E.P. 246,516, 25.1.26. Conv., 24.1.25).—The resistance to disintegration of the filament of a gas-filled electric incandescence lamp is reduced by the addition of a few hundredths of one per cent. of carbon monoxide to the inert gas, *e.g.* argon, contained in the lamp bulb, at two-thirds of an atmosphere pressure. J. S. G. THOMAS.

Electrodes for electric arc lamps. GEN. ELECTRIC Co., LTD., W. SINGLETON, and A. HARE (E.P. 248,187, 17.3.25).—Carbon electrodes are heated to about 700° for about 1 hr. in a stream of silicon fluoride vapour, and then cooled. When in use, an adherent, non-oxidisable coating forms on the hot parts of the carbon, and the rate of consumption is reduced. If required, the electrodes may be preheated under reduced pressure. J. GRANT.

Composition for storage-battery positive electrodes. R. C. BENNER, Assr. to PREST-O-LITE Co., INC. (U.S.P. 1,572,512, 9.2.26. Appl., 6.11.23).—Compositions containing lead peroxide and lead sulphate suitable for preparing active material for the positive electrodes of storage batteries, especially those of the type which initially do not require to be charged, yield when treated with alkali hydroxide solutions, *e.g.* 20% sodium hydroxide solution, crystalline compounds which serve as bonding compounds and facilitate the application of the material to the grids. The paste obtained hardens well when dried at 65°. T. S. WHEELER.

Electrolyser with stirring device. A. BARTH (G.P. 423,801, 15.1.24).—A horizontal diaphragm perforated in the centre has a cylindrical diaphragm fixed on it above the perforation, the joint being watertight. The reaction liquid can be circulated from the cathode space to the anode space, or in the reverse direction, by a pump, the diaphragm being used as a filter. A. B. MANNING.

Electrolytic cell. F. PETZ, Assr. to ELEKTRIZITÄTS-A.-G. VORM. SCHUCKERT & Co. (U.S.P. 1,579,138, 30.3.26. Appl., 18.7.24).—See E.P. 222,856; B., 1925, 511.

Process for producing ozone. A. STARKE and H. VON WARTENBERG, Assrs. to OZONHOCHFREQUENZ G.M.B.H. (U.S.P. 1,579,162, 30.3.26. Appl., 2.2.23).—See E.P. 193,422; B., 1923, 986 A.

See also pages 401, **Chlorinated products from cellulose waste lyes** (G.P. 419,815); **Utilising gases from cellulose digesters** (G.P. 423,198). 406, **Cyanides from cyanamide salts** (Austr. P. 101,316). 417, **Preservation of nitrogen compounds in manure** (G.P. 424,667). 419, **Lactose from whey** (E.P. 248,998).

XII.—FATS; OILS; WAXES.

Analysis of soya-bean oil. H. PFAHLER (Chem. Umschau, 1926, **33**, 65—70).—A freshly prepared sample of soya-bean oil was found to have the following composition: linolenic acid 1·9%, linoleic acid 29·2%, *iso*linoleic acid (including a little *iso*linolenic acid) 24·3%, oleic acid 30·8%, stearic acid 7·0%, palmitic acid 2·35%, glyceryl residue (as C_3H_2) 3·88%, and unsaponifiable matter 0·5%. These numbers do not differ much from those of Baughman and Jamieson (B., 1923, 149 A), and yield a theoretical iodine value of 130, which is in close agreement with the value 132·4 obtained experimentally. To prepare linoleic acid the mixed fatty acids were brominated and the tetrabromide was separated and converted into the methyl ester of linoleic acid. The acid liberated from the ester had iodine value 172·4 (theoretical 181·4). This linoleic acid was again brominated; the resulting tetrabromide (m.p. 113°) corresponded to only 33% of the original linoleic acid. It appears that in debrominating the bromide isomerisation of the acid takes place so that what is re-brominated is a mixture of isomeric linoleic acids leading to a solid bromide and to a liquid one. H. M. LANGTON.

China wood [tung] oil. H. WOLFF (Chem. Umschau, 1926, **33**, 70—72).—A critique of the work of Fonrobert and Pallauf (B., 1926, 371), and a restatement of the author's view that the thickening or gelatinisation of tung oil is colloidal in character. The results of molecular weight determinations are regarded as unimportant since such determinations in systems of high molecular weight are uncertain and high values may be due to the formation of molecular aggregates, and not necessarily proof that polymerisation has occurred. Marcusson has shown that tung oil in benzene solution thickens under the action of ferric chloride. A similar effect is produced by hydrochloric acid and sulphurous acid. The author allowed tung oil to thicken to the point of gelatinisation in the one case by heating at 200° and in the other by acting on it with 0·1% of hydrochloric acid solution (*d* 1·125). The viscosity and the iodine value of the two samples of oil were taken from time to time and the results plotted against time. The viscosity curves were nearly coincident, but the curves indicating fall in iodine value became widely divergent. Owing to the relatively long time occupied in the change from α- to β-elæostearin it appears unlikely that β-elæostearin crystals are formed in quite a short time during drying. Moreover the

characteristic phenomena associated with tung oil occur equally in strongly diffused light and in darkness where a change into the β-form cannot yet be assumed. H. M. LANGTON.

Tall oil, a by-product of sulphate-cellulose manufacture. M. DITTMER (Z. angew. Chem., 1926, **39**, 262—269).—Tall oil, obtained by hydrolysing the soap separating from black liquor from sulphate-cellulose manufacture, probably contains decomposition products of lignin and oxidised acids, in addition to the resin from the wood. The boiling process hydrolyses esters and the oil contains, therefore, only acids and unsaponifiable substances. A commercial sample from Sweden contained 12·4% of unsaponifiable substances, 30·4% of resin acids, 54·9% of fatty acids, and 2·2% insoluble in petroleum ether. This last substance imparts the dark colour to the oil and may be removed by dissolving the oil in petroleum ether, decahydronaphthalene, hexahydrotoluene, or hexahydroxylene. The unsaponifiable substances consist of a phytosterol ($C_{32}H_{38}O$) and hydrocarbons. The acids were separated into two classes by esterification in the presence of sulphuric acid; the usual procedure with hydrochloric acid cannot be used because the resin acids are slowly attacked by this acid. The resin acids are abietic acids ($C_{20}H_{30}O_2$); pinabietic acid (cf. Aschan, A., 1921, i, 669) is only obtained from the distillate from tall oil. The iodine value (Wijs) of the fatty acids is not constant, but depends on the time of treatment and the excess of halogen taken. This is due to polymerisation of unsaturated acids during the cellulose boiling process and to gradual depolymerisation during the determination of the iodine value; the molecular weight (284) calculated from the saponification value is less than that (369) found cryoscopically by Rast's camphor method. Similar abnormalities were observed when the fatty acids from herring oil were heated with alkali under pressure (Persapol process). The density of the fatty acids was normal (0·907—0·910). By catalytic hydrogenation only stearic acid was obtained, and the saponification value of the original acids also indicated a chain of 18 carbon atoms. No saturated acids were found in the original acids, but oleic, linoleic, and linolenic acids were identified. A. GEAKE.

Semi-micro-method for rapid determination of the iodine value [of fats and oils]. B. M. MARGOSCHES and E. NEUFELD (Chem.-Ztg., 1926, **50**, 210—211).—The sample (0·05—0·075 g. for fatty oils, and 0·1—0·2 g. for solid fats) is dissolved in 5 c.c. of alcohol at 50°, 10 c.c. of 0·2*N*-alcoholic iodine solution are added, followed by 10 c.c. of water, and, after shaking for 5 min., the excess of iodine is titrated with 0·05*N*-thiosulphate. The results compare favourably with those obtained by the macro-method. A. R. POWELL.

See also A., April, 358, **Equilibria underlying soap-boiling processes** (MCBAIN and ELFORD). 381, **Hydrogenation of organic substances at high temperature under pressure** (KLING and FLORENTIN).

PATENTS.

Increasing the durability of pure salad or sweet oils. H. BOLLMANN, Assr. to M. F. FOSTER (U.S.P. 1,575,529, 2.3.26. Appl., 28.5.25).—A small quantity of lecithin is added to the oil directly after purification. H. M. LANGTON.

Preparation of highly viscous lubricating oils. CONTINENTALE A.-G. FÜR CHEMIE (G.P. 423,702, 21.4.21).—Lubricating oils are prepared from mobile animal oils by heating them in a vacuum to about 300°, and maintaining them at that temperature for a long time. A. B. MANNING.

Oxidation of linseed oil (Swiss P. 101,617).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Evaluation of white pigments. A. EIBNER (Z. angew. Chem., 1926, **39**, 261—262).—A criticism of Wolski's paper (cf. B., 1925, 930) on this subject. A. GEAKE.

Transformation and oxidation of chromium sesquioxide. BLANC and CHAUDRON.—See A., April, 372.

Apparent paradox of plasticity. DE WAELE.—See I.

Tall oil. DITTMER.—See XII.

PATENTS.

Resinous condensation products. BRIT. THOMSON-HOUSTON CO., Assees. of J. G. E. WRIGHT and W. J. BARTLETT (E.P. 235,589, 12.6.25. Conv., 13.6.24).—The initial fusible resinous product from phthalic acid and glycerol is dispersed in a suitable medium, such as ethyl phthalate or benzyl benzoate, and heated to 210—290°. After this treatment the resinous product, recovered, *e.g.*, by precipitation, is non-corrosive to metals, and when further heated passes readily into the final insoluble condition without liberation of phthalic anhydride. D. F. TWISS.

Manufacture of water-insoluble resin soaps. R. F. UZAC (F.P. 595,497, 20.3.25).—An alcoholic solution of magnesium chloride or other alcohol-soluble metal salt is added to an alcoholic solution of an alkali resinate or an alcoholic solution of a resin neutralised with sodium alkoxide. The product is separated from sodium chloride by filtration and the alcoholic solution of a metal resinate thereby obtained is suitable for use as a lacquer. A. J. HALL.

Manufacture of staining solutions and lacquers from soft lignite. C. EHRENBERG, H. WIEDERHOLD, C. KRUG, M. G. HOLSBOER, K. FISCHER, and STUDIENGES. F. AUSBAU DER IND. (G.P. 422,739, 28.12.21).—Staining solutions for wood are obtained by extracting lignite with acetone or its homologues containing hydrochloric acid; the resulting stains have the advantage over those obtained by extraction with alkaline solutions in being fast to washing with water. Cellulose acetate may be

dissolved in the acetone solutions, and the products used as paints or lacquers. A. J. HALL.

Oxidation of linseed oil. P. W. H. J. V. DE WAEL (Swiss P. 101,617, 2.5.22).—Oxidising gases are led through linseed oil, the temperature of the gases being controlled so that the temperature of the oil does not exceed 300°. After passage through the oil, the gases are led through a condenser to recover vaporised fatty acids. A. J. HALL.

Increasing the durability of lacquers from cellulose (F.P. 595,208).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Utilisation of clotted latex for making sheet [rubber]. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1926, 8, 135—136).—The use of sodium silicofluoride (2 g.) with clotted latex (3000 c.c.) prevents further decomposition and enables coagulum and sheet to be prepared free from bubbles. In spite of the retarding effect of sodium silicofluoride the finished sheet rubber from clotted latex vulcanises more rapidly than the normal. D. F. TWISS.

PATENT.

Increasing the durability of products from cellulose (F.P. 595,208).—See V.

XV.—LEATHER; GLUE.

Solvents for tannins. MICHEL-DURAND (Compt. rend., 1926, 182, 937—939; cf. A., 1924, i, 477).—Boiling water is a very satisfactory solvent for extracting tannins from wood, and extracts as much tannin-material as combined extractions by means of acetone, alcohol, ether, water, etc. Only a portion of the water-soluble tannins is soluble in acetone. L. F. HEWITT.

PATENTS.

Drying gelatin. H. W. LUSCHER, Assr. to UNITED STATES GLUE CO. (U.S.P. 1,575,348, 2.3.26. Appl., 30.3.22).—Filtered air is, in part, passed through an ozoniser and the resulting air is mixed with the remainder which has been heated; the substantially sterile gelatin is submitted to the drying action of this mixture. D. F. TWISS.

XVI.—AGRICULTURE.

Nitrogen-fixing capacity of soils. WINOGRADSKY (Compt. rend., 1926, 182, 907—910).—The nitrogen fixation by soils from widely different sources has been investigated by the method described previously (cf. B., 1925, 328). In each case approximately the same amount of nitrogen (about 10 mg.) was fixed per 1 g. of mannitol consumed. With the most active soil 120 hrs. were required for the complete decomposition of 2 g. of mannitol under the conditions described. L. F. HEWITT.

Determination of the adsorption capacity and "degree of saturation" of soils. E. BOBKO and D. ASKINASI (Trans. Inst. Fertilisers [Russ.], 1925, Lief. 25, pp. 39; Chem. Zentr., 1926, I, 2045).—In place of the method of Gedroiz (leaching with ammonium chloride solution and analysis of the filtrate), the authors displace the bases originally present by means of barium chloride and then extract the adsorbed barium with 0·05*N*-hydrochloric acid or, in the case of chalky soils, with *N*-sodium chloride; barium only need then be determined. The results obtained are somewhat higher than by the ammonium chloride method. Repeated determinations on the same sample proved that the adsorptive capacity of a Podsol soil was not changed by this treatment; five experiments reduced it by about 12% only. On the other hand, the adsorptive capacity of black soils is greatly reduced—about 42% after four experiments. Ammonium chloride decomposes the soil in much smaller amount. C. T. GIMINGHAM.

Injurious action of heavy dressings of calcium carbonate on Podsol soils in connexion with the special character of the biological processes occurring therein. A. TJULIN (Trans. Inst. Fertilisers [Russ.], 1925, Lief. 26, pp. 39; Chem. Zentr., 1926, I, 2043).—Heavy dressings of calcium carbonate indirectly affect the biological processes in the soil unfavourably, influencing the nitrogen cycle in particular. In presence of harmful amounts of calcium carbonate, soluble nitrogen compounds, including nitrites, are produced in greater amounts and denitrification follows more rapidly than under other conditions. The alkalinity and the amount of humus soluble in water are increased in such soils. Addition of ammonium bicarbonate intensifies this action of calcium carbonate, whereas sodium or potassium bicarbonate tends to reduce it. Light "unsaturated" soils are specially sensitive towards calcium carbonate. C. T. GIMINGHAM.

Influence of calcium carbonate on the decomposition of organic substances in soil. A. TJULIN (Trans. Inst. Fertilisers [Russ.], 1923, Lief. 14, pp. 12; Chem. Zentr., 1926, I, 2042).—A comparison of the humus content of a slightly acid soil after treatment with different amounts of dung, green manure, and calcium carbonate. The soils receiving calcium carbonate were poorer in humus than the control soils. The loss in "humic acids," *i.e.*, that part of the humus which is extracted by 2·5% caustic soda solution but not by 0·5% sulphuric acid, was particularly marked. The material soluble in 0·5% sulphuric acid was increased by manuring with calcium carbonate. C. T. GIMINGHAM.

Liming and the nitrate of soils. N. REMESOV (Trans. Inst. Fertilisers [Russ.], 1925, Lief. 30, 105—117; Chem. Zentr., 1926, I, 2042).—Changes in the nitrate content of soils manured with different amounts of calcium carbonate and dung have been followed from July to October. On Podsol soils, neither calcium carbonate nor dung alone had much effect, but together they caused a large increase in nitrate. On light sandy soils, dung alone increased the nitrate content more than when calcium carbonate was also added. C. T. GIMINGHAM.

Fertilising action of calcium carbonate. E. TRUNINGER (Landw. Jahrb. Schweiz, 1925, 39,

807—842; Chem. Zentr., 1926, I, 2230).—The addition of calcium carbonate to acid soils has an injurious effect upon the action of simultaneously applied phosphatic fertilisers, but has no effect on their action when the soil has a neutral or alkaline reaction. The different action of calcium carbonate upon acid and non-acid soils is caused by the different adsorptive properties of the two types of soil, calcium carbonate having an injurious effect only when the soil is capable of adsorbing or decomposing it. Calcium carbonate should never be added in conjunction with phosphatic fertilisers to acid soils. The higher the acidity, the greater the risk of injurious effect by the addition of calcium carbonate. The high adsorption of hydroxyl ions by the soil colloids, and the presence of buffer substances protect plants from the harmful effects of excessive alkalinity of the soil solution.

L. A. COLES.

Trials with powdered phosphorite on different soils. K. KALININ (Trans. Inst. Fertilisers [Russ.], 1925, Lief. 30, 118—127; Chem. Zentr., 1926, I, 2041).—In pot experiments with oats, weathered black soils, which showed little response to manuring with nitrogen and potassium, gave increased yields of 50—70% with powdered phosphorite used at the rate of 0·15 g. of phosphoric acid per kg. of soil. On Podsol soils, where potassium and nitrogen give large increases of crop, manuring with phosphorite was ineffective.

C. T. GIMINGHAM.

Influence of manuring with calcium chloride on some crops. O. DAFERT and F. G. ERDÖDY (Fortschr. Landw., 1, 69—78; Chem. Zentr., 1926, I., 2041).—In pot experiments with mustard, oats, and beans, addition of calcium chloride increased the green weight but not the dry weight of mustard and oats and hastened the absorption of nutrients by mustard but not by oats. The yield of dry matter was reduced by calcium chloride in the case of beans. There was an increase in the chlorine content of the tissues of all plants.

C. T. GIMINGHAM.

Utilisation of the nitrogen of peat by plants. P. KUPREENOK (Trans. Inst. Fertilisers [Russ.], 1923, Lief. 19, pp. 15; Chem. Zentr., 1926, I., 2042).—Peat (Hochmoortorf) acted as a source of nitrogen and increased the yield of oats when given with potassium phosphate and chloride. Its utilisation was facilitated by addition of calcium carbonate and the introduction of nitrifying bacteria with dung. The plants utilised 20—28% of the nitrogen of the peat.

C. T. GIMINGHAM.

Effect of straw on plant growth. R. C. COLLISON and H. J. CONN (New York Agr. Exp. Stat., 1925, Tech. Bull. 114, 35 pp.).—Vegetation experiments with wheat, barley, and buckwheat confirmed the injurious effect upon the plants of addition of straw and other highly carbonaceous plant residues to the soil or sand. The effect is especially marked if the supply of available nitrogen is low and it can be corrected by addition of nitrate. In a heavy soil, injury did not appear until growth had proceeded for several weeks, but in sand cultures, the harmful influence of straw or a water extract of straw was evident soon after germination. Seedlings also showed injury at a very early stage in water culture, even under sterile conditions or when the solutions were changed very frequently. It is generally accepted that abundance of highly carbonaceous material stimulates development of micro-organisms which compete with the plants for the available nitrogen, but this can hardly account for the effects upon quite young seedlings. It is suggested that a second factor is involved—the presence of toxic compounds in the straw. Analyses indicated the presence of salicylic acid, dihydroxystearic acid, and vanillin, all known to be toxic to plants.

C. T. GIMINGHAM.

Is the potato an acid-sentitive plant? M. TRENEL (Die Kartoffel, 1926, [3], 28—30; Chem. Zentr., 1926, I., 2042).—Potatoes show vigorous growth in soils of p_H 4·0—6·0. Injury appears below p_H 4·0 and in soils of alkaline reaction.

C. T. GIMINGHAM.

Influence of [seed treatment with] magnesium chloride and mercury-containing materials on plant yield. D. MEYER (Deuts. Landw. Presse, 1924, 51, 461—462; Chem. Zentr., 1926, I., 2044). —The so-called seed-stimulating solutions are discussed in connexion with "seed-pickling" solutions. 2·5% magnesium chloride solution acts unfavourably on the germination of oats, but not of barley. "Uspulun" and "Germisan" have little effect on germination. Pot and field experiments with oats, wheat, and barley gave no increased yields due to treatment of the seed.

C. T. GIMINGHAM.

Adherence to foliage of sulphur in fungicidal dusts and sprays. R. W. THATCHER and L. R. STREETER (New York Agr. Exp. Stat., 1925, Tech. Bull. 116, 18 pp.).—Determinations are recorded of the amount of sulphur remaining on apple foliage after treatment with sulphur dusts and "lime-sulphur" sprays. During the first week after treatment, 89—94% of the sulphur applied as dust was lost from the leaves; in the same time, the loss of sulphur derived from "lime-sulphur" spray was from 45 to 75%. Addition of calcium caseinate to the spray fluid slightly reduced this loss. After this rapid mechanical loss during the first days, the decrease in the amount of sulphur adhering proceeded slowly and fairly regularly. The larger loss of sulphur from the dust application is attributed to the large size of the particles as compared with those derived from the decomposition of the polysulphides of "lime-sulphur." C. T. GIMINGHAM.

Concentration of phosphorite ore. I. VERCHOVSKI (Trans. Inst. Fertilisers [Russ.], 1924, Lief. 22, 75 pp.; Chem. Zentr., 1926, I., 2040).—By heating phosphorite ore to 500—600° before grinding, the cohesion between the phosphorite concretions and the matrix is, to a great extent, broken down and the mass becomes more friable. A mechanical

concentration of the phosphorus-containing part of the mineral can thus be obtained.
C. T. GIMINGHAM.

Determination of perchlorate in Chile saltpetre. LEIMBACH. HAHN.—See VII.

PATENTS.

Fertiliser containing urea. COMP. DE L'AZOTE ET DES FERTILISANTS S.A. (F.P. 600,016, 23.6.25. Conv., 26.6.24).—A mixture of calcium cyanamide with a solution of a salt, *e.g.*, potassium sulphate, containing an acid radical capable of precipitating calcium, but in quantity insufficient to precipitate the whole of it, is heated under atmospheric or increased pressure, preferably after removal of the precipitated calcium salt. The solution, which contains urea and potassium hydroxide, is saturated with carbon dioxide and evaporated to dryness, or is used in the treatment of further quantities of calcium cyanamide. L. A. COLES.

Storing mixed fertilisers containing ammonium nitrate. J. HEPPES (G.P. 423,806, 24.12.24).—Caking of the fertilisers is prevented by storing them at a temperature below 5°.
L. A. COLES.

Preservation of organic nitrogen compounds in liquid manure. L. KUCHLER and J. BODLER (G.P. 424,667, 23.1.23).—Fresh farm or stable manure, in the form of liquid or paste, is subjected to the action of an alternating current, and when completely sterilised is stored in tightly closed pits.
L. A. COLES.

XVII.—SUGARS; STARCHES; GUMS.

See A., April, 386, **Occurrence of crystallised lævulose** (VON LIPPMANN). 387, **Molecular weight of soluble starch** (PICTET). 441, **Pectins of the sugar-beet** (EHRLICH and VON SOMMERFELD).

PATENTS.

Decolorising and defecating product (U.S.P. 1,575,561).—See II.

Treating bagasse (U.S.P. 1,574,254).—See V.

Lactose from whey (E.P. 248,998).—See XIX.

Glutamic acid (E.P. 248,453).—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Valuation of hops. W. WINDISCH, P. KOLBACH, and W. BANHOLZER (Woch. Brau., 1926, 43, 79—82).—In the valuation of hops by expert examination and handling, the visual estimate of the amount of lupulin present is supposed to give a rough measure of the richness of the hops in bitter principles. Working on 14 samples of hops from different districts, the authors have compared the visual estimates of lupulin-content with the results of analytical determinations of the bitter acids and resins by the most recent methods, and found no correspondence whatever. J. H. LANE.

Wort boiling under pressure. P. PETIT and J. RAUX (Fond. Brass. et Malterie, 1925, Bull. 4, 62; Woch. Brau., 1926, 43, 29—30).—The effect of excess pressures up to 1 atm. during wort boiling was studied on a wort made from malt with 12·5% of maize and rice, by a two-mash process. The wort was treated with 100—150 g. of hops per hl., two-thirds being added at the commencement of boiling and the remainder ½ hr. before the end. Compared with boiling under atmospheric pressure the higher pressures were found to permit a better utilisation of hops and to produce an earlier and better break or flocculation of proteins. On the other hand they intensified the colour of the wort and to a smaller extent that of the fermented beer, and tended to produce an unpleasantly bitter flavour. Better results were obtained by boiling for a short time at relatively high excess pressures, such as 1 atm., than for a longer time at lower ones; thus the intensification of colour was much less in ¼ hr. under 1 atm. than in 1 hr. at 0·1—0·5 atm. above the normal. J. H. LANE.

Influence of sugar on determination of ammonia in grape must. J. VENTRE and E. BOUFFARD (Compt. rend., 1926, 182, 784—786).—In presence of lævulose or, to a less extent, of dextrose (but not sucrose), ammonia cannot be completely removed from aqueous solution by the usual distillation process. To determine ammonia in must therefore, it is necessary to distil to dryness with magnesia in a vacuum. R. CUTHILL.

See also A., April, 431, **Enzymic breakdown and synthesis of carbohydrates** (VON EULER and MYRBÄCK). 432, **Activity and iron content of highly active catalase preparations** (HENNICHS). 433, **Yeast maltase. Separation of maltase and invertase** (WILLSTÄTTER and BAMANN). 434, **Effect of hydrogen cyanide on alcoholic fermentation** (WARBURG); **Effect of hydrogen sulphide on chemical processes of the cell** (NEGELEIN); **Alcoholic fermentation in presence of hydrogen sulphide and hydrogen cyanide** (NEUBERG and PERLMANN); **Growth of yeast in wort** (LUDWIG). 435, **Lactic acid fermentation** (VIRTANEN, KARSTRÖM, and BÄCK); **Fermentation of α-ketoglutaric acid by** ***Bact. xylinum*** (IWATSURU).

PATENTS.

Preparing yeast for use in food products. C. HOFFMAN and C. N. FREY, Assrs. to FLEISCHMANN Co. (U.S.P. 1,575,762, 9.3.26. Appl., 15.9.23).—A product containing ammonium sulphate and devitalised yeast in which the protoplasmic material in the cells has been coagulated, is obtained by treating yeast with saturated ammonium sulphate solution at 85°, washing out at least a portion of the ammonium sulphate, filtering, and drying the residue in such a manner that the cell walls are not ruptured.
L. A. COLES.

Fixation of solar or ultra-violet energy (E.P. 226,534).—See VII.

Glutamic acid (E.P. 248,453).—See XX.

XIX.—FOODS.

Reagent for detection of peroxydase in milk. P. BORINSKI (Z. angew. Chem., 1926, 39, 281—283).—0·85 g. of guaiacum resin is dissolved in 85 c.c. of 70% alcohol, and 10 c.c. of phenol and 5 c.c. of 3% hydrogen peroxide are added. The reagent may be prepared in 1 hr., can be used immediately, and is stable for at least a week. Milk no longer gives a blue colour with this reagent after heating for 30 min. at 70°, 3 min. at 75°, or 1 min. at 85°; the colour is given if 10% of unheated milk is added. A. GEAKE.

Action of streptococci on milk. C. GORINI (Compt. rend., 1926, 182, 946—947).—Streptococci may be divided into two classes, the one class (saprophytic), occurring in bovine excreta, clot milk which has been thoroughly sterilised until of yellow tint, giving rise to a large amount of lactic acid and yielding a clot which is not digested readily; the other class (parasitic), found on udders and in bovine and human infections, will only clot milk which has been mildly sterilised, do not produce much lactic acid, and the clot formed is readily digested by acids. L. F. HEWITT.

Composition of fenugreek seeds and their admixture with wheat used for flour-milling. E. FLEURENT (Compt. rend., 1926, 182, 944—946).—Fenugreek imparts a disagreeable bitter flavour to flour when mixed with it, even to the extent of 0·01%, and the bread made from the flour is unsaleable. This effect is due to the fact that fenugreek seeds contain about 9·5% of a brown oil, smelling of fennel, d^{18} 0·97, iodine value 110—114, extracted by ether, and a resin (13·5%) with a bitter taste, extracted by alcohol. L. F. HEWITT.

Determination of the age of bread. J. P. PEPER (Chem. Weekblad, 1926, 23, 163—168).—The ageing of bread is accompanied by a shrinkage in volume of the starch grains and loss of water; these phenomena are inhibited by presence of acetaldehyde. The method devised by Katz consists in kneading a weighed quantity of the crumb with water through silk gauze, making up to a known volume in a measuring cylinder, adding toluene and acetaldehyde, and noting the height of the column of sediment after 24 hrs. This height is compared with readings obtained from breads of known age. It is found possible in this way to determine whether a bread is quite fresh or more than 12 hrs. old. S. I. LEVY.

Tin-plate for containers for food products (preserves etc.). H. SERGER (Chem.-Ztg., 1926, 50, 201—202).—The most frequent source of trouble in tin-plate to be used for food containers is local porosity due to the presence of particles of slag, dirt, etc., accidentally introduced into the iron before or during the rolling process. These particles absorb acid during the pickling, and bubbles result when the sheet is tinned, which ultimately form porous spots on the plate. Exposed iron, which may readily be detected by the potassium ferricyanide–gelatin method, is due to incomplete removal of oxide scale from the sheet during the pickling process, with the result that tin is not deposited on these spots. The chemical composition of the iron is of considerable importance in obtaining a satisfactory tin-plate. B. W. CLARKE.

[Decomposition of] pectins. A. MEHLITZ (Konserven-Ind., 1926, 13, [2], 1—3; Chem. Zentr., 1926, I., 2154; cf. B., 1925, 687).—If apple-pectin and pectin-sugar solutions are heated for 10 hrs., 16% and 19·4% of their pectin-contents respectively are destroyed, mostly in the first few hours. In the latter case the decomposition is increased and decreased by the presence of sugar and acid (produced in the initial stages) respectively, the high final value being due to the higher boiling point of the solution. When the duration of boiling is less than 2 hrs. the decomposition of pectin is appreciably less in presence of sugar. J. GRANT.

Relative nutritive value of various proteins contained in Japanese food articles. U. SUZUKI, Y. MATSUYAMA, and N. HASHIMOTO (Sci. Papers Inst. Phys. Chem. Res., 1925, 4, 1—48).—By feeding experiments on rats, the relative nutritive values of the proteins of various kinds of fish, rice, wheat, soya beans, kaoliang, Indian corn (maize), barley, oats, rye, peanut, cotton seed, banana, and potato have been studied. A table is given showing the different effects of the various proteins on the growth of rats. H. J. CHANNON.

Preventing bumping in the determination of crude fibre [in feeding stuffs etc.]. W. LEPPER (Chem.-Ztg., 1926, 50, 211).—In the determination of crude fibre in material containing large amounts of mineral constituents, the preliminary boiling with sulphuric acid presents no difficulties, but when the insoluble material from this operation is collected on asbestos filters and the fibre and asbestos subsequently boiled with potassium hydroxide there is a marked tendency for the liquid to bump. This may be avoided by just covering the bottom of the beaker with glass beads 5 mm. in diameter. These may subsequently be separated by decantation or by screening on a sieve with 3 mm. holes. A. R. POWELL.

See also A., April, 428, **Maintenance values for proteins of milk, meat, bread and milk, and soya-bean curd** (ROSE and McLEOD). 436, **Biological assay of fat-soluble vitamins** (CHICK); **Maintenance of standardised breed of rats for work on fat-soluble vitamins** (SMITH and CHICK). 437, **Vitamin-B of lemon rind** (WILLIMOTT); **Use of colloidal ferric hydroxide for adsorbing vitamins-B and -D** (ZAJDEL and FUNK); **Water-soluble vitamin content of velvet bean** (SALMON and MILLER); **Action of *n*-butyl nitrite on activated cholesterol and vitamin-D** (BILLS); **Effect of irradiation on antirachitic properties of milk** (STEENBOCK and others); **Antirachitic value of fresh spinach** (CHICK and ROSCOE); **Antirachitic value of winter spinach** (BOAS).

441, Alcohol-soluble protein from polished rice (Hoffman). 444, Determination of calcium in milk (Corley and Denis); Determination of protein nitrogen (Voit); Determination of pectin (Emmett and Carré).

Patents.

Obtaining milk sugar [lactose] from whey. A. G. Bloxam. From Elektro-Osmose A.-G. (Graf Schwerin Ges.) (E.P. 248,998, 17.7.25).—Whey is neutralised, *e.g.*, with sodium carbonate, boiled for a short time, and then, while cooling, is gradually acidified, *e.g.*, by addition of strong hydrochloric or sulphuric acid. After filtering off precipitated albumins, the solution is decolorised and further quantities of the albumins are removed by stirring it with about 1—2% of an adsorbent, such as clay or kaolin, and again filtering. Electrolytes and other diffusible impurities are subsequently removed by electro-osmotic treatment under such conditions that losses of lactose by diffusion and inversion are reduced to a inimum. For example, the liquid may be subjected to the action of a continuous electric current in a narrow compartment between anode and cathode diaphragms constructed respectively of animal membranes, such as skin, bladder, or gut, and woven vegetable material, such as cotton cloth, the temperature being kept below 40°. Lactose of 96—97% purity is obtained on evaporation of the solution *in vacuo* or by spraying, and pure lactose can be obtained from it by crystallisation. L. A. Coles.

Desiccating apparatus [for milk and buttermilk]. N. P. Collis, Assr. to Collis Co. (U.S.P. 1,575,414, 2.3.26. Appl., 14.1.22).—A desiccating apparatus for milk and buttermilk comprises a steam-heated rotating drum beneath which is a pan containing the liquid to be desiccated. The buttermilk is pumped from the pan and sprayed through slots on to the drum where it is desiccated and from which the dried material is removed in the course of a rotation. Any excess of liquid is returned from the spray pipe to the pan, the contents of which it serves to agitate. A second pump and spray system are provided for use with sweet milk and as this dries more rapidly than buttermilk it is delivered to the drum nearer the scraping point. Gearing is provided by which either pump can be coupled with the driving engine. T. S. Wheeler.

Making concentrated sour milk. A. Neuhauser (U.S.P. 1,576,351, 9.3.26. Appl., 19.1.24).—Skim milk is concentrated to about one-fourth of its bulk, and the product is soured at about 32—38° to an acidity of over 4%, and is then homogenised. L. A. Coles.

Self-preserving stock food. W. P. M. Grelck (E.P. 249,746, 9.9.25).—See U.S.P. 1,554,913; B., 1925, 939.

Recovery of solid constituents of liquids (E.P. 248,081).—See I.

Preparing yeast for use in food products (U.S.P. 1,575,762).—See XVIII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Iodoeosin as indicator in alkaloidal determinations. D. B. Dott (Pharm. J., 1926, **116**, 357).—As an indicator for the titration of strychnine, iodoeosin is inferior to cochineal, hæmatoxylin, or methyl-orange. E. H. Sharples.

Alleged deterioration of Indian opium on keeping. D. B. Dott (Pharm. J., 1926, **116**, 356).—Examination at intervals over a period of two years, of a sample of Indian opium which had been dried at 95—99° and preserved in a stoppered bottle in the dark, demonstrated that the morphine content decreased by only 0·24%. E. H. Sharples.

Benzylmorphine tartrate. D. B. Dott (Pharm. J., 1926, **116**, 356).—The solubilities in water of the neutral tartrate (containing 1 mol. H_2O) and of the sulphate of benzylmorphine, are 1 in 3 and 1 in 170 pts. respectively. E. H. Sharples.

Determination of small amounts of alcohol and ether vapours in the presence of each other. E. von Somogyi (Z. angew. Chem., 1926, **39**, 280—281).—Alcohol vapour is absorbed in a mixture of 3 pts. of water and 1 pt. of concentrated sulphuric acid by vol., and ether vapour in a mixture of equal volumes of concentrated sulphuric acid and *N*-potassium dichromate. Ether retained by the first absorbent is drawn into the dichromate by sucking air through the apparatus for 3 hrs., and the dichromate reduced measures the ether content. The alcohol is also determined by oxidation with dichromate. A. Geake.

Adsorption from solution and the valuation of adsorbents used in therapy. T. Sabalitschka and W. Erdmann (Pharm. Ztg., 1926, **71**, 374—376, 390—392).—Experiments on the adsorption of barium chloride, ferric chloride, basic aluminium acetate, potassium hydroxide, hydrochloric acid, oxalic acid, acetic acid, sulphosalicylic acid, nicotine, methylene-blue, tannin, and iodine by kieselguhr, boneblack, barium sulphate, lime-, beech-, and animal-charcoals, and charcoal sponge ("Schwammkohle") indicate that the order of the adsorptive powers of the adsorbents for the above compounds is not uniform. Animal charcoal, charcoal sponge, and boneblack generally possessed the highest, and barium sulphate the lowest adsorptive powers. Boneblack had the highest adsorptive power for bases, but was inferior to lime- and animal-charcoals and charcoal sponge for the adsorption of acids. The present knowledge of the application of adsorbents in therapy is discussed and it is concluded that generalisations regarding the behaviour of adsorbents in the animal body deduced from experiments *in vitro*, are only valid within very narrow limits owing to the predominating effect of exterior conditions on the adsorptive power. E. H. Sharples.

See also, A., April, 408, **Existence of *iso*pulegone in nature** (GRIGNARD and SAVARD); **Terpene and sesquiterpene of Mitsuba-zeri** (HIRANO). 409, **Purification of digitonin** (WINDAUS). 417, **Conversion of berberine into β-homochelidonine (α-allocryptopine)** (HAWORTH and PERKIN, JUN.). 430, **Relation between chemical constitution and taste of sweet substances** (TÄUFEL). 440, *Chlorocodon whiteii*: **its constituents and their harmacological actions** (DILLING). 444, **Micro-determination of lactic acid and lactates** (HANSEN)

PATENTS.

Manufacture of acridine derivatives [bactericides]. BRIT. DYESTUFFS CORP., W. H. PERKIN, and A. W. BURGER (E.P. 248,182, 12.3.25).—2:8-Diamino-3:7-dimethoxy- (or -ethoxy-)acridine derivatives or the corresponding 3 : 7-2 : 8-compounds prepared by standard methods, and the acridinium compounds derived from them by acetylation, alkylation, and hydrolysis, have bactericidal properties, the acridinum compounds being especially suitable for therapeutic use, owing to their greater solubility. 2:8-Diamino-3:7-diethoxyacridine, m.p. 281°, is prepared by heating a mixture of 42·5 pts. of 2:4-diaminophenetole, 35 pts. of oxalic acid, 40 pts. of zinc chloride, and 45 pts. of glycerin, to 200° in 1½ hrs., and maintaining it at that temperature for 2 hrs. When cool, the product is ground and extracted for several hours with cold, dilute ammonia, and, after filtration, the acridine derivative is extracted from the residue with boiling alcohol, and recovered by evaporation or by precipitation with dilute ammonia; it is purified by crystallisation from pyridine and water. 2:8-Dimethoxy-3:7-diaminoacridine: a mixture of 1 pt. of sodium nitrite with 34·2 pts. of 3:3′-diacetamido-4:4′-dimethoxydiphenylmethane, m.p. 159—160°, prepared by the acetylation of 3:3′-diamino-4:4′-dimethoxydiphenylmethane (cf. F.P. 322,985; B., 1903, 414), is added to a mixture of 68 pts. of sulphuric acid, *d* 1·7, and 21·6 pts. of nitric acid, *d* 1·49, at 0—5°. After stirring for 2 hrs., during which the temperature rises to 15—20°, the mixture is poured on to 2000 pts. of ice and water, and the dinitro-compound, m.p. 260—261°, is recovered by filtration, and hydrolysed by heating with strong sulphuric acid on the water bath to 6:6′-dinitro-3:3′-diamino-4:4′-dimethoxydiphenylmethane, m.p. 226—228°. A solution of 17·4 pts. of the product in 207 pts. of hydrochloric acid, *d* 1·185, is treated with 21·5 pts. of zinc dust, heated under a reflux condenser for 24 hrs., diluted with an equal volume of water, boiled, and oxidised with ferric chloride; the acridine compound is then salted out and crystallised from water.

L. A. COLES.

Manufacture of glutamic acid and salts thereof. K. IKEDA (E.P. 248,453, 3.12.24).—Glutamic acid is recovered from vinasses or other beet sugar residues in the form of its calcium salt, $C_5H_7O_4NCa,H_2O$, either by digestion of the liquor with lime or, preferably, with an acid hydrolytic agent, followed by neutralisation with lime. Separation of the sparingly soluble calcium salt is facilitated by addition of alcohol or a soluble calcium salt and ammonia. Alternatively, glutamic acid may be separated from the acid hydrolysis product in the form of its hydrochloride, which is converted into the calcium salt by treatment with lime. From the calcium salt, by addition of mineral acid, glutamic acid may be obtained, or, by treatment in solution with an alkali salt of an acid which forms insoluble calcium salts, soluble alkali glutamates can be prepared. The soluble calcium salt, $Ca(C_5H_8O_4N)_2$, can be obtained by treating the sparingly soluble salt with carbon dioxide in presence of water.

E. H. SHARPLES.

Manufacture of monoacyl derivatives of aminoarylarseno-compounds. G. NEWBERY, and MAY AND BAKER, LTD. (E.P. 248,523, 6.1.25).—Monoacylamidoarylarseno-compounds are prepared by combining equimolecular proportions of an arylarsenic compound and an acylamidoarylarsenic compound. Thus, an arylarsenious oxide or an aryldichloroarsine is condensed with an acylamidoarylarsine, or an acylamidoarylarsenious oxide or an acylamidoaryldichlorarsine with an arylarsine. For example, 3-acetamido-3′-amino-4 : 4′-dihydroxyarsenobenzene (light yellow powder) is prepared by condensing 3-acetamido-4-hydroxyphenylarsenious oxide with 3-amino-4-hydroxyphenylarsine hydrochloride in 10% sodium hydroxide solution. The compounds are also prepared by reduction of an arylarsinic acid and an acylamidoarylarsinic acid in admixture. For example, 3-benzamido-3′-amino-4 : 4′-dihydroxyarsenobenzene is obtained by reducing a mixture in equimolecular proportions of 3′-amino-4-hydroxyphenylarsinic acid and 3-benzamido-4-hydroxyphenylarsinic acid in dilute sodium carbonate solution with sodium hyposulphite and magnesium chloride. E. H. SHARPLES.

Manufacture of pure urea [carbamide]. J. Y. JOHNSON. From BADISCHE ANILIN & SODA-FABR. (E.P. 249,041, 5.11.25).—Pure carbamide is prepared by treating concentrated solutions of technical carbamide at moderately elevated temperatures and in presence of at least 0·5—1% of ammonia with an oxidising agent leaving no soluble residues, preferably with air, oxygen, or manganese dioxide, and separating from the precipitated impurities. Compounds of the heavy metals are precipitated practically entirely in a form in which they can be easily separated. After separation, a clear colourless solution is obtained from which, by crystallisation or complete evaporation, pure, white carbamide is produced. E. H. SHARPLES.

Producing chloro-derivatives of methane. C. B. CARTER and A. E. COXE, Assrs. to S. KARPEN & BROS. (U.S.P. 1,572,513, 9.2.26. Appl., 9.8.23).—A mixture of 100 vols. of chlorine, 100 vols. of methyl chloride, and 200 vols. of methane is passed into a chamber at about 450°, when chlorination of the methane with formation of 80% of methylene chloride, 15% of chloroform, and 5% of carbon tetrachloride rapidly occurs, all the chlorine being

consumed. The issuing vapours are washed in a tower with water to remove hydrochloric acid and then compressed to 10—15 atm. to condense methylene chloride, chloroform, and carbon tetrachloride. The condensate is distilled and any methane and methyl chloride evolved are returned to the uncondensed gases which, after expansion, are mixed with 100 vols. of chlorine and 46 vols. of methane and passed again to the reaction chamber. The process is continuous and all danger of explosion is obviated. T. S. WHEELER.

Extraction process [for concentration of acetic acid]. H. SUIDA (F.P. 594,925, 10.3.25).—In a continuous process for concentrating aqueous solutions of substances volatile in steam, *e.g.*, acetic acid, the dilute solution is heated under pressure in a still and the vapours are led into the base of an extraction column down which trickles a boiling solvent not readily miscible with water. Acetic acid vapours are absorbed by the solvent, which is drawn off at the base of the column. The solution then passes to a second still which is heated at a slightly lower pressure by hot vapours from the top of the preceding column, and the operations are repeated in a succession of stills and columns, the pressure in the final still being atmospheric. R. B. CLARKE.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Intensification by dye-toning. A. and L. LUMIÈRE and A. SEYEWETZ (Brit. J. Phot., 1925, **73**, 147—148; Bull. Soc. Franç. Phot., 1926, iii., **13**, 5—8).—The silver image is mordanted in a bath containing copper sulphate 40 g., tripotassium citrate 60 g., glacial acetic acid 30 g., ammonium thiocyanate (dissolved separately) 20 g., water 1 litre. After washing, the image is dyed in a mixture of 1% solutions of Methylene Blue (287 c.c.), Rhodamine S (333 c.c.), and Phosphine M, patent extra concentrated (380 c.c.), with addition of 1% acetic acid. This bath gives a neutral coloured image, and an intensification greater than that obtained by other means. The intensified image can be reduced by a solution of potassium permanganate 1 g., sulphuric acid 5 c.c., water 1 litre, or by hyposulphite which, however, alters the colour. The colour of the image can be altered by various modifications to obtain different results in printing. W. CLARK.

PATENTS.

Dye composition [for treating photographic emulsions]. E. J. WALL and D. F. COMSTOCK, Assrs. to KALMUS, COMSTOCK AND WESCOTT (U.S.P. 1,573,595, 16.2.26. Appl., 6.7.17. Renewed 12.9.23). —A composition for treating photographic emulsions contains a protective emulsoid colloid for preventing the coagulation of the suspended dye. For example, with an isocyanine dye a fraction of 1% of gelatin may be used. E. S. KREIS.

Treating photographic emulsion, and colour-sensitising composition. E. J. WALL and D. F. COMSTOCK, Assrs. to KALMUS, COMSTOCK AND WESCOTT (U.S.P. 1,573,596, 16.2.26. Appl., 11.7.17. Renewed, 12.9.23).—The sensitising composition contains an oxidising agent to prevent reduction of the emulsion by the protecting agent for the colloidal dye, and an alkali to prevent bleaching of the dyestuff. Hydrogen peroxide is a suitable oxidising agent. E. S. KREIS.

Photographic medium. M. C. BEEBE and A. MURRAY, Assrs. to WADSWORTH WATCH CASE CO. (U.S.P. 1,574,359, 23.2.26. Appl., 18.11.22; cf. E.P. 203,285; B., 1924, 493).—The medium consists of a hydrophobic colloid with lead triethyl iodide as sensitiser. W. CLARK.

Photographic sensitiser. M. C. BEEBE and A. MURRAY, Assrs. to WADSWORTH WATCH CASE CO. (U.S.P. 1,575,143, 2.3.26. Appl., 18.11.22).—The sensitiser consists of a hydrophobic protective colloid, containing a dispersed halogen-liberating compound. W. CLARK.

Light-sensitive layers. B. STANGE (G.P. 421,138, 18.10.21).—The layer contains silver halide particles stained in three primary colours. It consists of organic particles, such as bacteria (yeast cells, acetic bacteria), dyed in the appropriate colours, and treated first with a silver salt solution and then with a solution of a halide. W. CLARK.

Toning sulphide-toned silver images on development and printing-out papers. LEONAR-WERKE ARNDT & LÖWENGARD (G.P. 422,295, 5.8.24, and 422,296, 18.9.24. Addns. to 413,360; B., 1925, 787).—Thiocarbamide, thiosinamine, alkali thiocyanate, sodium thiosulphate, or mixtures of these are added to simple or slightly complex gold or platinum salts, with or without the addition of lead salts, and either in the solid state or in solution. The images may be toned with selenium as well as with gold. W. CLARK.

Selenium toning bath for developing and printing-out papers containing silver. LEONAR-WERKE ARNDT & LÖWENGARD (G.P. 422,297, 11.11.24. Addn. to 419,428).—The selenium is dissolved in hyposulphite solution in presence of alkaline reacting substances other than sodium carbonate, *e.g.*, sodium sulphite, ammonia, organic ammonium bases, caustic soda, or caustic potash. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Pressure wave sent out by an explosive. I. W. PAYMAN and H. ROBINSON (Safety in Mines Res. Bd., Paper No. 18, 1926, 60 pp.).—As part of an investigation of the effect of the detonation of mining explosives on the atmosphere surrounding a borehole, the waves sent through air by different types of explosion have been photographed by the "Schlieren" (striæ) method devised by Töpler

(Ann. Physik, 1867, **131**, 33). The apparatus and technique used in the present investigation are fully described. The waves were set up by the "explosion" produced when a celluloid disc was caused to burst under gaseous compression, by the pressure and shock waves from a gaseous detonating mixture, and by the explosion of a low-tension detonator. A series of 48 photographs of atmospheric shock waves are reproduced, and a non-mathematical explanation is given of their initiation and propagation. A method is also described for obtaining an accurate record of the rate at which the shock wave sent out by an explosive is propagated through the atmosphere, the length and duration of the flame produced, and the relative positions of shock wave and flame at any instant. An appendix contains a summary of other methods which have been proposed for investigating these waves.

S. Binning.

Patent.

Explosives. A. C. Scott, and Mexco, Ltd. (E.P. 248,089, 29.11.24 and 19.8.25).—The particles of an explosive mixture containing a combustible substance and a compound containing readily available oxygen are coated with a synthetic resin, *e.g.*, a phenol–aldehyde condensation product. By using a liquid or plastic resin the explosive will be rendered plastic. A gel of a phenol–formaldehyde resin and a nitro-compound may be used as a coating. The phenol–aldehyde condensation product may form the combustible constituent of the mixture. Explosives made in this way are waterproof and may be grained.

S. Binning.

XXIII.—SANITATION; WATER PURIFICATION.

Control of the aluminium sulphate process for the purification of drinking water. F. Egger (Chem.-Ztg., 1926, **50**, 167).—The determination of the sulphate content of the water before and after the clarification process gives a more accurate indication of the efficiency of the precipitation than the determination of the aluminium, which gives unsatisfactory results. The process may be represented $Al_2(SO_4)_3+3Ca(HCO_3)_2=3CaSO_4+2Al(OH)_3+6CO_2$ and owing to the corrosive nature of the carbon dioxide, the amount of this left in the water must be carefully controlled by milk of lime, the acidity of the filtered water as indicated by phenolphthalein being a satisfactory guide to the efficiency of the removal. The determination of the hydrogen-ion concentration is the most accurate method of control. The determination of organic impurities and bacteria enables the filtration process itself to be controlled.

B. W. Clarke.

Effect of temperature on rate of deoxygenation of diluted sewage. R. E. Greenfield and A. L. Elder (Ind. Eng. Chem., 1926, **18**, 291—294).—The rate of deoxygenation of sewage samples diluted with aerated distilled water and incubated at temperatures of 20° and 14° is fairly adequately represented by the formulæ connecting rate, time, and concentration developed by Theriault, Streeter, and Phelps (cf. B., 1925, 565), and others; but this is not the case at lower temperatures (2—6°), chiefly on account of the occurrence at these temperatures of a lag phase during the first few days of incubation when little or no oxygen is absorbed. The lag phase, the duration of which decreases with increase in sewage concentration, is attributable to the slow development at low temperatures of the bacteria required to carry on the deoxygenation. Biochemical oxygen demand curves for diluted sewage give no indication of two stages of deoxygenation such as those obtained by Theriault for Ohio River water. Such curves were obtained, however, from samples of Illinois River water. The two-stage phenomenon seems to be one encountered in river water but not always in diluted sewage. It is suggested that the two stages, rather than representing carbonaceous oxidation and nitrification (cf. Adeney, B., 1896, 463), indicate first, the decomposition of the dead organic matter contained in the river water, and second, the decomposition of dead plankton which at the time of collection of the sample are living organisms.

W. T. Lockett.

Treatment of packing-house sewage. W. C. Moor and W. P. Wayne (Ind. Eng. Chem., 1926, **18**, 239—242).—In 1916 an experimental activated sludge plant was constructed at Fort Worth, Texas, for the treatment of packing-house waste. With an aeration period of 10 hrs. clear and stable effluents were obtained, but the cost of aeration was excessive. The present system consists of treatment by screens and Dorr clarifiers, the resulting partially purified waste being then passed to the city sewers. About 2½ million gallons of stock-yard waste are treated per day. Screening removes 24% of the suspended solids and 27% of the grease content of the waste; the Dorr clarifiers, which are provided with grease skimmers, remove a further 50% of the suspended solids and 49% of the grease. By the entire process (screens and clarifiers) a total of 98% of the suspended solids (as determined by settling for 2 hrs.) is removed. The city disposal plant comprises screens, grit chambers, Imhoff tanks, sprinkling filters, Dorr clarifiers for the retention of humus, and drying beds for sludge.

W. T. Lockett.

Patent.

Processes involving exchange reactions, particularly softening of water by zeolite-like bodies. E. B. Higgins (E.P. 248,414, 1.10.24).—Instead of passing the water through a bed of the softening material, the softening material is suspended in the water, by giving the water an upward flow. Various forms of apparatus are described. For example, by causing the water to flow upwards in a vessel like an inverted cone, the softening material in one zone will rise with the water, in another it will just keep in suspension, and in the uppermost zone it will settle through the water. Regeneration and washing of the material are effected in a similar manner. By this method larger quantities of water can be treated in a shorter time than by the old method, owing to the more intimate contact achieved and greater surface of softening material exposed.

E. S. Kreis.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 11, 1926.

I.—GENERAL; PLANT; MACHINERY.

Practical applications of the Lewis filtration equation. H. C. Weber and R. L. Hershey (Ind. Eng. Chem., 1926, 18, 341—344).—The Lewis filtration equations are of the form, $V=KP^{1-s}$ and $V=K'\theta^{(1+t)/(2+t)}$ for constant rate and constant pressure conditions respectively, where V=volume of filtrate, P=pressure, θ=time, and s and t are dependent on the nature of the cake and the operating conditions. Observation of these practical conditions in the filtration of defecated sugar syrups by Sweetland presses shows that these equations can be most usefully applied to decide whether the requisite amount of filter aid is being used. The value of s obtained by plotting the logarithm of V against the logarithm of P should be zero if sufficient filter aid is being used to prevent the formation of a compressible cake. The value of t will show whether scouring or plugging of the cake is taking place. The complexity of the filtering process, however, makes exact measurements very difficult, since it is clmost impossible to keep the operating conditions aonstant throughout a run. B. W. Clarke.

Absorption of gases by a liquid drop. W. G. Whitman, L. Long, jun., and H. Y. Wang (Ind. Eng. Chem., 1926, 18, 363—367).—The absorption of carbon dioxide by a drop of water falling freely through a column of the gas, treated as a case of liquid film diffusion, gives the coefficient for the average rate of absorption per c.c. of solution, k_L as 260 g. of carbon dioxide per hour through 1 sq. cm. of interface with unit pressure. The absorption of ammonia by the drop considered as an example of gas film diffusion gives the coefficient, k_G, as 22 g. of ammonia under unit driving potential expressed as partial pressure of ammonia in atmospheres, this figure being confirmed by the value 18 determined from the humidification of air by the drop. Although these rates are higher than those obtained by other methods, *e.g.*, absorption through flat liquid surfaces, from gas bubbles, or from columns with wetted walls, the ratio k_L/k_G is about the same as that obtained by the other methods; this shows that the spray type of equipment is as suitable for slightly soluble gases as the bubble type. B. W. Clarke.

Foam meter. H. E. Williams (Ind. Eng. Chem., 1926, 18, 361—362).—A mechanically operated egg beater is fixed in a bowl-shaped receptacle with an outlet at the lowest level, and the liquid under examination is poured in to cover the beater blades. After stirring for a definite period (3 min.) the foaming liquid is immediately run out so as completely to fill a weighed flask under the outlet. The volume of the flask is termed the foam meter volume (F.M.V.), and tho foaming tendenoy of paints, varnishes, etc., may be numerically expressed as the ratio of weight of material placed in the bowl at first to the weight of the F.M.V. of material after whipping. The apparatus may also be used to test the froth-reducing powers of anti-frothing oils. D. G. Hewer.

Increasing the internal volume of silica gels. Holmes, Sullivan, and Metcalf. See VII.

Patents.

[Fritted quartz] filters. Jenaer Glaswerk Schott & Gen. (E.P. 226,182, 22.11.24. Conv., 11.12.23. Addn. to 218,286, B., 1925, 230).—A filter of fritted particles of either natural quartz or broken fused quartz is constructed in a similar mamer to the glass filter described in the original patent. B. M. Venables.

Edge filtration. H. S. Hele-Shaw and J. A. Pickard (E.P. 249,202, 17.12.24).—Paper for use in the edge filters described in E.P. 210,101, 210,376, and 227,890 (B., 1924, 239; 1925, 232) is treated with rubber, chlorinated rubber, celluloid, casein, cellulose esters, synthetic or natural resins, paraffin, hardened gelatin, or asphaltum, with the object of preserving the effectiveness of the filter medium even when this is wiped or scraped to discharge the residue. The substance need only be applied to the filtering edges of the paper and is applied after cutting the sheets to shape, and either before or after assembling into packs. B. M. Venables.

Filter. D. M. Goetschins and L. F. Vogt, Assrs. to Standard Chemical Co. (U.S.P. 1,569,627–8, 12.1.26. Appl., 25.8.22).—(a). A filter designed especially for the filtration of corrosive liquids without the use of filter-cloth comprises a number of units, each of which consists of a fine-grain porous stone mounted in a frame. The units are arranged one above the other, a filter chamber being formed between adjacent filter-stones. Each stone has a bevelled corner portion which serves as a filling port. Outlet passages for the filtrate are disposed vertically in the side portion of each unit. (b). Each unit of the filter consists of a frame and a series of filter-stones arranged edge to edge in the plane of the frame. The filter-stones of adjacent units form a filter chamber between their opposed side faces. Means are provided for feeding mixed liquids and solids to the chamber. The chambers discharge through outlets in the inner edge portions of the stones into outlet passages for the filtrate disposed between the inner opposed edges of adjacent filter-stones. R. B. Clarke.

Filter. G. J. PEZOLD (U.S.P. 1,574,409, 23.2.26. Appl., 19.12.23).—A horizontal cylinder contains a filtering screen which is attached to the cylinder where a horizontal plane through the axis cuts the sides, and is dished downwards almost to touch the bottom. A conveyor and scrapers to remove the solid from the screen and means for treating the solid with water, air, or steam are provided. The cylinder has a removable head at one end to allow of easy access to the screen. T. S. WHEELER.

Filtering and thickening process and apparatus. A. L. GENTER (U.S.P. 1,575,907, 9.3.26. Appl., 11.4.21).—A mixture passes through a filtering medium, and the flow of the filtrate is periodically reversed by changing the respective levels of the mixture and the filtrate. The solids accumulated on the medium are thus discharged into the mixture and thicken it. H. MOORE.

Filter unit. J. J. PREBLE, Assr. to SPRAY ENGINEERING Co. (U.S.P. 1,576,121, 9.3.26. Appl., 30.4.25).—A filter cell is formed of a densely-compacted stack of variously-shaped expanded metal sheets disposed between inlet and outlet openings. H. HOLMES.

Filtration and treatment of liquids. C. V. ZOUL, Assr. to CELITE Co. (U.S.P. 1,579,171, 30.3.26. Appl., 13.2.23).—Before using a filtering apparatus for filtration proper a clear liquid is repeatedly circulated through it, and to the liquid a filter aid is added gradually so as to form a bed of filter aid upon the filtering surfaces. B. M. VENABLES.

Ovens or furnaces. H. B. CANNON (E.P. 234,049, 18.12.24. Conv., 14.5.24).—The hearth of the furnace is made of a material with high heat-radiating qualities, *e.g.*, silicon carbide. Above the hearth is a heating chamber containing combustion chambers also made of material with high heat-radiating qualities and communicating with a flue situated below the hearth, the bottom of the flue possessing high heat-absorbing qualities. The heat from the combustion chambers is radiated to the arched roof of the heating chamber and thence to the hearth, which is protected by a screen from direct radiant heat from the combustion chambers. The furnace is specially suitable for firing enamelled ware. B. W. CLARKE.

[Roasting] furnace. H. H. BUBAR (U.S.P. 1,576,033, 9.3.26. Appl., 10.8.23).—Material to be treated in a roasting furnace is delivered through a preheater on to one end of an extended stationary hearth and is moved along the hearth by a rabble. Means are provided for passing the gases from the material around the preheater out of contact with the material in the preheater. H. HOLMES.

Furnace. F. SEEBER (U.S.P. 1,576,371, 9.3.26. Appl., 30.3.25).—An annular hearth is mounted for rotation in a closed annular chamber. Partitions extending from the roof of the chamber into proximity to the hearth define with the roof and side walls of the chamber a closed chamber for charging material to and discharging it from the hearth. A combustion chamber connected with the furnace chamber is arranged at one side of the charging chamber, a flue communicates with the furnace chamber at the opposite side of the charging chamber, and a gas-tight seal is provided between the hearth and the walls of the furnace chamber. H. HOLMES.

Vertical cone mills for grinding dry granular products. J. H. GENTRUP (E.P. 242,614, 21.10.25. Appl., 5.11.24).—The apparatus comprises an outer rotating vertical cone, and an inner fixed cone, the large ends of the cones being downwards and the space between the two being wider at the top than the bottom, and being adjustable by altering the vertical position of the inner cone. B. M. VENABLES.

Pulverising or grinding mills. J. B. C. SCHERBAUM (E.P. 245,097, 15.12.25. Conv., 24.12.24).—A disc grinding mill is constructed so that the periphery only of the grinding discs is enclosed in a casing which may be maintained under reduced pressure, the larger part of the surface of the discs being left exposed to the air for cooling. The casing bears resiliently but tightly against the rotating disc, and the grinding surfaces of the discs consist of intercalating ridges and grooves which decrease in depth towards the periphery. B. M. VENABLES.

Grinding and the like mills. C. V. GREENWOOD (E.P. 249,631, 28.1.25).—Grinding is effected by the action of a number of discs (driven by a spider) against the interior surface of a tyre or ring. The discs are constructed of laminae preferably disposed at a slight inclination to the plane of rotation, and the outer grinding ring is formed of a number of detachable and renewable bars arranged like the staves of a tub, alternate bars being preferably of hard and soft metal. B. M. VENABLES.

Comminuting solids and forming colloidal solutions. O. AUSPITZER (U.S.P. 1,577,052, 16.3.26. Appl., 23.7.25).—To prevent waste of power in internal friction in a colloid mill, the material issues from the mill at a point directly behind that at which the beaters exert their action and is then conveyed outside the mill back to the point of disintegration through an external pump. Preferably the material is caused to flow in a straight confined tangential course through the mill. E. S. KREIS.

Mill. F. SILVA (U.S.P. 1,577,376, 16.3.26. Appl., 21.11.25).—A rotary casing is provided with external trackways and supporting rollers. Spaced supports project through feed and discharge openings at opposite ends of the casing. Crushing rollers are carried by the supports within the casing, and means are provided for forcing the supports downwards, these means yielding if undue resistance is met, and for maintaining a constant fluid level within the casing. H. HOLMES.

Softening of hard calcareous materials such as water-laid deposits and incrustations [scale]. A. A. COZENS, and GAS LIGHT AND COKE Co. (E.P. 248,886, 28.1.25).—Hard calcareous deposits from water are broken down by treatment with a solution

of free citric acid until a soft mass or sludge is formed which can be removed by flushing. The disintegration may be effected with application of heat. The citric acid may have a concentration of 10—30% or more and may contain 2—3% of an alkali chloride.
S. Binning.

Heating of vacuum pans. Blair, Campbell, and McLean, Ltd., and A. Blair (E.P. 248,897, 9.2.25).—Vacuum pans are heated by two or more pipes in the form of a coil the convolutions of which are in the same plane, each of the pipes being independently connected with the steam supply pipe and being provided with a discharge outlet for condensed water. Alternatively the pipe coils may be in pairs, the outer and inner ends of each pair being coupled together and connected to the steam supply pipe and discharge pipe respectively.
S. Binning.

Drying furnaces.—G. G. Brookway (E.P. 249,381, 1.7.25).—A drying furnace for powdered material, such as fuller's earth, boneblack or other filtering material is constructed of a number of vertical shafts down which the material trickles against hot gases issuing from burners inserted through the walls at the bottom of the shafts. The material is made to take a zig-zag path by blocks of refractory material projecting from the walls and extending about halfway across the shafts, the treated material finally emerging through nozzles of refractory material at the bottom. B. M. Venables.

Drying or calcining. J. A. Barr, Assr. to Internat. Agricultural Corp. (U.S.P. 1,573,533, 16.2.26. Appl., 9.4.24).—An apparatus for drying or calcining consists of a shaft down which the material under treatment is passed. Hot flue-gases are introduced at the bottom and are withdrawn by suction at the top of an intermediate zone in the shaft. Air drawn in at the bottom of the shaft by the suction in the exit flue cools material which has passed out of the heating zone, and air similarly drawn in at the top of the shaft prevents any moisture passing up from the heating zone and softening untreated material so that it chokes the shaft.
T. S. Wheeler.

Apparatus for concentrating solutions [by freezing]. M. O. Johnson (U.S.P. 1,576,136—7, 9.3.26. Appl., 19.9.21).—(a) A dilute solution to be concentrated is cooled by heat exchange with the concentrated juice leaving the apparatus, and is then partially frozen in a refrigerator which is cooled at the upper part only by circulating brine. The remaining liquor or concentrated juice collects in the lower part of the refrigerator and leaves through the heat exchanger (cf. *supra*). The brine is cooled in a brine tank by the expansion of a refrigerant vapour, the latter is then compressed, partially cooled by ordinary means (*e.g.*, water sprays), further cooled by the ice obtained from the juice, and expanded again. All the heat exchanges are effected without mixing the various fluids. (b) In a scheme similar to that described in (a) the refrigerator is provided with means such as a worm conveyor for continuously scraping off and removing the ice formed, which is then centrifuged and the separated liquor returned to the refrigerator. The brine after leaving the refrigerator proper is used to cool the liquor in the feed tank. B. M. Venables.

Absorption apparatus. C. L. Voress, V. C. Canter, and R. W. Skoog, Assrs. to Gasoline Recovery Corp. (U.S.P. 1,577,200, 16.3.26. Appl., 3.8.25).—A perforated distributing pipo extends the entire length of a horizontal drum in its lower portion. A mechanical support is secured to the drum slightly above the distributing pipe, and carries stationary solid absorbent material with the interposition of a second mechanical support provided with openings sufficiently small to prevent the passage of particles of the absorbent material. A second perforated pipe extends along the upper portion of the drum above the absorbent material. H. Holmes.

Charcoal absorption apparatus. Farbw. vorm. Meister, Lucius, & Brüning, Assees. of R. Tiedtke and J. Benz (G.P. 421,027, 29.8.24).—The patent relates to improvements on the apparatus described in G.P. 377,520 and 378,608 (*cf.* E.P. 187,223 and 191,005; B., 1923, 386a; 1924, 237). An annular chamber containing charcoal rotates above a second annular chamber divided into two circular compartments, the planes of contact of the chambers being so perforated that the perforations can be brought into or out of alinement. Gas passes into the outer compartment of the lower chamber, then through perforations into the upper chamber; any unabsorbed gas is led out through the inner compartment. R. B. Clarke.

Air-cooled still. E. C. Ballman, Assr. to J. H. Cassidy (U.S.P. 1,577,675, 23.3.26. Appl., 19.1.20).—The still comprises a lower boiling vessel open at the top, surmounted by an upper condensing vessel closed at the top, but open at the bottom. A small gutter is provided around the interior of the condenser to catch the condensate.
B. M. Venables.

Regulating the temperature of superheated steam. Farbenfabr. vorm F. Bayer & Co., Assees. of K. Hencky (U.S.P. 1,578,313, 30.3.26. Appl., 7.11.24).—The superheated steam is passed downwards at high velocity over a thin film of water also flowing downwards and spread over an extensive area of surface. B. M. Venables.

Multiple-deck pulp thickener. C. W. Ankeny, Assr. to Dorr Co. (U.S.P. 1,578,625, 30.3.26. Appl., 26.4.22).—The apparatus comprises a number of superposed settling compartments having central openings for the downward discharge of settled solids and openings connecting adjacent compartments for the upflow of supernatant liquid. Scraping devices pass over the bottom of each compartment and immediately adjacent thereto in order to deliver settled solids towards the discharge opening.
J. S. G. Thomas.

Apparatus for effecting the transfer of heat between gases. [Recuperators.] T. TEISEN (E.P. 249,919, 31.12.24).

Powdered fuel [boiler] furnaces. C. HOLD (E.P. 250,103, 18.9.25).

Dryers, mixers, screeners and the like. E. E. WELCH (E.P. 249,998, 18.3.25).

Separation of liquids of different specific gravities. B. D. COMYN and W. A. WHITE (E.P. 250,360, 10.2.25).

Silicious alkaline-earth product (U.S.P. 1,574,363).—See VII.

Calcium silicate derivative (U.S.P. 1,574,380). —See VII.

Electric gas purifier plants (E.P. 240,844).—See XI.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Limits of inflammability of firedamp in atmospheres which contain blackdamp. H. F. COWARD and F. J. HARTWELL (Safety in Mines Res. Bd., Paper No. 19, 1926, 11 pp.).—The limits of inflammability of methane in atmospheres composed of air with additions of nitrogen and carbon dioxide have been determined at atmospheric temperatures and pressures. Two series of experiments were carried out, with tubes of 2 in. and 10 in diam. respectively, and upward propagation of flame. The differences observed with the two tubes were almost inappreciable. The upper and lower limits of inflammability approach one another and finally coincide as the proportion of diluent is increased. Flame propagation is not possible with methane mixed with air containing more than 25% of carbon dioxide or 38·5% of nitrogen, or intermediate percentages of blackdamp, depending on the proportions of nitrogen and carbon dioxide in the latter. The upper limit of inflammability of methane falls rapidly as a diluent gas is added to the air. The lower limit rises comparatively slowly in atmospheres containing up to 20% of diluent gas, showing that the addition of moderate amounts of either nitrogen or carbon dioxide to the air would not appreciably diminish the danger of a firedamp explosion underground. A. B. MANNING.

Electric ignition of firedamp: alternating and continuous currents compared. R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 20, 1926, 18 pp.).—The minimum current required in order that the spark or "momentary arc" produced when the circuit is broken should cause ignition of a mixture of methane and air has been determined under a variety of conditions. Three types of apparatus were used in which the contact between two metallic poles could be broken automatically every 5 sec., the poles remaining in contact for 0·5 sec. The break of circuit in one apparatus was similar to that occurring during underground signalling, and in the others to the sudden break of a wire under tension. The poles were enclosed in a glass vessel which could be evacuated and filled with the gas mixture under investigation, which in most of the experiments contained about 8% of methane. The igniting current, for a given mixture of methane and air, varies with the volatility of the metal at the spark-gap, being lower the lower the b.p. of the metal. Other conditions being the same, the igniting current is lower the more rapid the break of circuit and the smaller the area of contact at the moment of break of circuit. The relation between igniting current and inductance of the circuit varies with the mechanical conditions under which the sparks are produced. Under one set of conditions the relationship could be expressed by the equation $Li^{1\cdot4}=k$. In general, for low voltages, particularly with high self-inductive circuits, the current is of far greater importance than the impressed voltage as regards the igniting power of the spark. With higher voltages, however, the igniting current increases. The character of the current supply, whether continuous or alternating, does not materially affect the igniting power of the spark, the results obtained in this series of experiments differing from those recorded by Thornton (Trans. Roy. Soc., 1914, A **90**, 272). A. B. MANNING.

Simplified method of evaluation of coals. F. LANDSBERG (Glückauf, 1925, **61**, 1427—1439; Chem. Zentr., 1926, I, 2271—2272).—The method, which is used by the German State Railways, depends on the relation between calorific value and content of volatile constituents. The cal. val. rather than the quantity of the volatile hydrocarbons is determined, and by plotting the cal. val. of the clean coal against its volatile content (or the volatile content reckoned as percentage against the cal. val. per 1% of volatile matter) curves are obtained which enable coals of various origins to be classified.

W. T. K. BRAUNHOLTZ.

Aspects of coke-oven heating in the light of modern tendencies in design. R. W. DAVIES (Gas World, 1926, **84**, Coking Sect., 38—42).—A heat balance for a 50-oven battery of standard design taking a charge of 8¼ tons of dry coal is given. The possible economies by the adoption of the most modern designs are estimated as follows. With an oven 40 ft. by 16 in. by 12 ft. 10 in. (charge 14½ tons) the radiation losses would be reduced from 160 B.Th.U. per lb. of coal to 92 B.Th.U. The use of insulating brick would bring this latter figure down to 61 B.Th.U. The chimney loss could be reduced by one-third by restricting the air supply, a part of the waste gases being returned to the ovens so that the total bulk of gases circulating and the flame conditions are not altered. The heat loss through open doors is reduced by reducing the pushing time. The total effect of these improvements would be the raising of the thermal efficiency from 46·0% to 66·2%, or a saving of fuel gas equivalent to 350 B.Th.U. per lb. of coal.

C. IRWIN.

Behaviour of coke-oven gas at low temperatures. W. H. NAYLOR (Gas World, 1926, 84, Coking Sect., 42—45).—Coke-oven gas stripped of carbon dioxide, hydrogen sulphide, and moisture was passed at the rate of 0·5—1·0 cb. ft. per hr. through tubes cooled with liquid air. 10 c.c. of condensate per cub. ft. of gas were obtained, containing on an average 79% of methane and 16% of ethylene. By fractional evaporation these constituents can be readily separated. C. IRWIN.

Origin of carbon disulphide in the carbonisation of coal. W. J. HUFF (Ind. Eng. Chem., 1926, 18, 357—361).—Coal of high sulphur content can be carbonised in the laboratory without producing carbon disulphide if it is heated slowly and gasification proceeds freely. Carbon disulphide apparently obtained in laboratory experiments by the interaction of coal gas and coke has been shown to be derived from the reheated coke alone; carbon disulphide presumably is not produced as the result of a secondary reaction between carbon and hydrogen sulphide When coal is heated rapidly so that a dense coherent coke is produced, *i.e.*, under conditions approximating to gas-works' practice, so that the tarry and gaseous vapours are only in contact with the heated coke for a relatively short time, considerable quantities of carbon disulphide are produced. Carbon disulphide is therefore a primary product of carbonisation, formed by rapid heating of the coal under conditions where local deficiencies of carbon and hydrogen exist, and the disulphide formed is rapidly removed from the hot coke. This offers an explanation for the low proportion of carbon disulphide produced in processes with a low rate of heat transmission, *e.g.*, low temperature carbonisation, vertical retorts, and coke-ovens, compared with horizontal gas retorts. B. W. CLARKE.

Solution of common salt as a confining liquid for gas analysis. H. TROPSCH (Z. angew. Chem., 1926, 39, 401; cf. Hoffmann, B., 1926, 146).—The addition of a small amount of sulphuric acid to solutions of salt used as confining liquid for gas analysis makes no appreciable difference to the solubility of carbon dioxide therein, but serves to neutralise traces of alkaline absorption liquids with which it becomes contaminated, and which otherwise would cause appreciable absorption of carbon dioxide. If the acidified solution is saturated with carbon dioxide and then left for some time exposed to the air, the amount of carbon dioxide it will subsequently absorb from gases containing a relatively high proportion of that constituent is inappreciable. To detect readily any change of reaction of the solution addition of a few drops of phenolphthalein is recommended. A. B. MANNING.

Experiences in benzol recovery by the vacuum method. GAREIS (Gas- u. Wasserf., 1926, 69, 269—272).—The Raschig system of benzol recovery (cf. B., 1923, 390 A) is characterised by the following features :—(1) The enriched oil is preheated only by the stripped oil leaving the still; (2) distillation is effected by indirect steam under high vacuum; (3) motor benzol is produced continuously without redistillation. The enriched oil is brought to 130° in the preheater (a tall narrow tube of wrought iron) and only requires a further heating to about 155° in the still. The evacuation of the still also tends to steam economy, the consumption being 3 kg. of steam per kg. of benzol against 6—10 kg. in older installations. The use of indirect steam produces a great saving in cooling water, the consumption being 35—60 litres per kg. of benzol. The deterioration of wash oil is very slow, one batch in the plant described having served for 21 months. This also appears to be due to the exclusion of direct steam. The cost of steam, cooling water, wash oil, and power for pumps per metric ton of benzol is estimated at M.47 against M.107 in the old process. The rotary benzol washer used is described and it is pointed out that the capacity of a washer depends not only on efficient contact of the gas and oil but on the efficient stripping of the latter, its temperature, and its mol. wt. Lignite oil was found unsatisfactory and coal-tar creosote low in naphthalene was used. With this oil naphthalene was by no means completely extracted from the gas. The stripped wash oil passes from the heat exchanger to the cooler at 35°, this low temperature facilitating separation of oil and water. A continuous plant for the treatment of benzol with acid and alkali was unsuccessful and was abandoned. C. IRWIN.

Autoxidation of mineral oils and determination of the tar value. J. MARCUSSON and W. BAUERSCHÄFER (Chem.-Ztg., 1926, 50, 263—264).—The acids produced in the sludge test of mineral oils have been examined. Those from a normal, unrefined transformer oil (d^{20} 0·891, open flash pt. 154°, acid value nil, tar value 0·88, ash 0·015%, viscosity at 20° 3·09), had a density greater than 1 and the copper salts were completely soluble in benzine. Treatment with benzine caused a separation into 60% of soluble, oily acids and 40% of insoluble, asphaltic acids. The soluble acids had acid value 66, saponif. value 130, acetyl value 58, iodine value 16, mean mol. wt. (Rast) 285. No reaction was given in the formolite test. The insoluble acids had acid value 67, saponif. value 269, acetyl value 127, iodine value 18. It is concluded that the soluble acids consist of hydroxynaphthenic acids formed from saturated naphthenes and that the insoluble acids are produced from unsaturated hydrocarbons by union of two molecules and the addition of oxygen. Of the acids from a refined white oil (d^{20} 0·842, open flash pt. 181°, free acid nil, sulphur 0·015%, tar value 14·5, viscosity at 20° 3·98), 90% were soluble in benzine and this fraction had d 1·014, acid value 121, saponif. value 240, acetyl value 44, iodine value 16, mean mol. wt. 357, and gave a negative formolite reaction. The copper salts were soluble in benzine and the peroxide content was very slight, the amount of active oxygen present being 0·06% (cf. Brauen, B., 1914, 951). A modified sludge test is described, wherein, by the use of sodium hydroxide as catalyst, a saving in time and oxygen is effected. 50 g. of the oil are mixed with 10 g. of

pumice saturated with sodium hydroxide (prepared by addition of the pumice to 0·7 g. of sodium hydroxide in 10 c.c. of water and drying at 105°) in a 200-c.c. conical flask and heated at 120° for 24 hrs. The mixture is cooled, 50 c.c. of 50% alcoholic sodium hydroxide (containing 7·5% NaOH) are added, and the liquid is heated under a reflux condenser for 20 min. The soap solution is separated, acidified, and extracted with benzene. From the extract, after removal of mineral acid by washing, the tarry matter is obtained by evaporation. This method, with refined transformer oils, gives results differing by only a few hundredths of 1% from those obtained by the standard German method. E. H. SHARPLES.

Decomposition of ozokerite by cracking. E. TOPORESCU (Bull. Acad. Sci. Roumaine, 1926, **10**, [2], 1—3).—See B., 1925, 980.

Chlorine in the petroleum industry. L. BURGESS (Trans. Amer. Electrochem. Soc., 1926, 49, 157—160. Advance copy).

PATENTS.

Drying of coal and other solid bituminous substances of the character of coal. KOHLENVEREDLUNG GES. M. B. H., and C. GEISSEN (E.P. 249,774, 17.11.25).—The moist material is brought into contact with the steam-heated walls of a dryer, hot inert gases being simultaneously passed through the material for the purpose of carrying away the vapours produced. W. T. K. BRAUNHOLTZ.

Manufacture of fuel. E. R. SUTCLIFFE (E.P. 249,170, 22.9.24).—Hard, homogeneous and stone-like briquettes are produced by compressing coal and lime which have been ground to pass a 200-mesh screen and have been intimately mixed in either the dry or the wet state. No binding agent is added to the mixture before briquetting, nor is the mixing carried out in such a way as to develop hydration of the lime or to render the mass plastic or more easily consolidated. The briquettes are carbonised and, after cooling and quenching, are broken into pieces of suitable size. The fuel so obtained is hard, dense, and uniformly porous, and it may be used as a substitute for charcoal. The sulphur which it contains is fixed by the lime and remains in the ash after burning. A. W. HOTHERSALL.

Block or briquette of lignite. E. FERNHOLTZ, Assr. to FERNHOLTZ MACHINERY CO. (U.S.P. 1,577,902, 23.3.26. Appl., 22.12.24).—The lignite is crushed, aged by storage for a limited time, dried by indirect heat derived from steam, cooled and simultaneously mixed and fed as a uniform mass into a mould of the extrusion type. The high pressure attained in the constricted mould causes the bitumen in the material to melt forming a strong waterproof binder. The briquettes are delivered from the mould without shock, and are fed gently along a channel until they have set and cooled.
A. W. HOTHERSALL.

Utilising coke contaminated with vegetable matter derived from the treatment of house refuse. G. H. HADFIELD (E.P. 249,247, 6.1 and 24.3.25).—The mixture of coke, coal, and vegetable matter obtained after treating house refuse in a liquid separating medium is dried, crushed, and briquetted, with or without the addition of small coal, coke, or anthracite duff. A. W. HOTHERSALL.

Carbonisation of coal. INTERNAT. COMBUSTION ENGINEERING CORP., Assees. of W. RUNGE (E.P. 242,622, 26.10.25. Conv., 6.11.24).—Finely-pulverised coal is introduced into the top of a vertical retort, and is carbonised in suspension, at a temperature of about 600—800° by an upward stream of hot gases produced by the partial combustion of the material at the bottom of the retort by means of a limited supply of air. The volatile products of carbonisation are removed at the top of the retort and the semi-coke at the bottom.
W. T. K. BRAUNHOLTZ.

Apparatus for carbonisation of fuel. S. R. ILLINGWORTH, ILLINGWORTH CARBONIZATION CO., R. DEMPSTER AND SONS, and H. J. TOOGOOD (E.P. 244,837, 25.9.24. Addn. to 245,190; B., 1926, 228).—The apparatus previously described, in which a system of vertical metal retorts is heated by radiant heat from flues in the walls of the setting surrounding the retorts, is modified by the addition of horizontal cross-over flues at right angles to the wall flues, and passing between the retorts. The hot gases pass through ports in the cross-over flues and come into direct contact with the retorts, which are thus heated by conduction as well as by radiant heat. The passage of the hot gases from the wall flues into the cross-over flues is controlled by dampers. Supplementary burners are provided in the wall flues directly over the cross-over flues. The upper ends of the retorts communicate with a header and are held by rings and preferably trough plates above the rings, and the lower ends have flanges of such a size resting on a base-plate that the retorts are free to be withdrawn through the header.
A. B. MANNING.

Producing combustible gases and coke from bituminous fuel. POWER GAS CORP., LTD., and N. E. RAMBUSH (E.P. 248,518, 30.12.24. Addn. to 235,958).—A modification of the main patent (B., 1925, 661) in which a portion of the mixed gas leaving the top of the shaft generator during the cold-blowing period is, after being scrubbed, returned to the bottom of the generator to replace part or whole of the steam used during that period, thereby producing gas of increased heating value.
A. B. MANNING.

[Prevention of corrosion in the] manufacture of fuel gases. C. COOPER, D. M. HENSHAW, and W. C. HOLMES & CO., LTD. (E.P. 248,841, 11.12.24).—Fuel gases are partially dried by being brought into intimate contact with a water-absorbing material in liquid form, *e.g.*, a solution of calcium chloride. The solution passes from the absorbing chamber to a heater, where the absorbed water is expelled, and the regenerated solution is returned to the chamber.

By this means the moisture content of a gas may be sufficiently reduced to prevent deposition of water in the mains, and the corrosive action of certain of the gas constituents is minimised or inhibited.

A. B. Manning.

Carbonisation of fuel by vertical retorts and the like. J. Rude (E.P. 248,967, 21.5.25).—A number of vertical retorts, arranged in concentric circular rows, are surrounded by cylindrical shell walls forming flues of which the inner wall accompanies the retorts in an intermittent rotary movement around a hollow axis, through which the distillation gases are withdrawn, whilst the outer shell is stationary and contains openings for the entry and exit of the heating gas. The waste gases pass through heat exchangers where they give up their heat to a gaseous medium (*e.g.* the fuel gas, or air) which goes to preheat the fuel to be carbonised. The rotary movement of the retorts serves to bring them in succession to the place where the discharge of the coke into a common cooling or quenching chamber takes place and where the recharging from a common hopper is effected. The connexions between the rotary and stationary parts of the apparatus are rendered gastight by means of liquid seals.

W. T. K. Braunholtz.

Fuel distillation. Merz & McLellan, and W. A. Riley (E.P. 249,236, 30.12.24).—The distillation of fuel is accomplished by injecting into the retort a mixture of hot gases produced by the combustion of producer or other gas in a special chamber, low-pressure steam which is superheated by contact with these combustion gases, and air which is admitted in regulated quantity to the combustion chamber in which the producer gas is burnt. The amount of admixed air is such that the required limited degree of combustion of fuel in the retort is attained (cf. E.P. 223,387, B., 1924, 1004). The steam is introduced by an injector nozzle either into the combustion chamber or into the conduit leading from the latter to the adjacent or superimposed retort, and it supplies the energy necessary to force the gases into the retort.

A. W. Hothersall.

Carbonising apparatus. O. H. Hertel (E.P. 249,710, 15.6.25).—The retort may be heated in stages progressively by separately controlled burners. It is provided with a central hollow shaft, which may carry a screw conveyor. The shaft is heated internally and progressively in stages by separately controlled burners. The pipes carrying the fuel gases to the internal burners are cooled, *e.g.*, by jackets in which air, steam, or water circulates. Firebrick may be interposed to prevent the play of the flame on the wall of the hollow member.

R. A. A. Taylor.

Water-gas generator with regenerative heat-storing device. M. Frankl (E.P. 240,466, 23.9.25. Conv., 26.9.24).—In the walls of a water-gas producer, on opposite sides of the central chamber, are two heat accumulators consisting of sets of vertical tubes open at the lower ends and connected by a manifold at the upper ends. During one gas generating and blowing period the tubes of one accumulator are heated internally by the "run" gases, and externally by the "blow" gases, and during the succeeding period the second accumulator is so heated. The heat stored is utilised for the generation of the steam necessary for gas production, by injecting water alternately into the tubes of the two heat accumulators. The steam and air are blown alternately in opposite directions horizontally through the fuel bed.

A. B. Manning.

Production of water-gas. Humphreys and Glasgow, Ltd., Assees. of C. S. Chrisman (E.P. 242,237, 19.8.25. Conv., 31,10.24).—Approximately one-half of the total water-gas is produced by supplying low-temperature steam to the generator fuel bed in a direction opposite to the flow of the air blast, removing the comparatively cool blue water-gas (*e.g.* at about 180°) from an outlet below the fuel bed, and passing it directly to the wash-box or to the holder. The remainder of the gas is produced by an up-steaming operation and is passed directly to the carburettor, which has been previously heated by combustion of the blast gases assisted by secondary air, and thence through the superheater to the wash-box or to the holder, where it may be mixed with the down-run gas to produce illuminating gas. In this way the carburettor may be kept at a high and comparatively uniform temperature, and economy is effected by having an unobstructed connexion between generator and carburettor without the usual hot-valve and reverse steam provisions. Where low-temperature steam is used in the generator, and blast products of low temperature are produced, the secondary air supplied to the carburettor may be preheated in order to accelerate the ignition of the blast gases. The heat carried away by the blast gases and by the carburetted up-run gas may be recovered by passing these gases through a recuperator, which serves to preheat the air or steam supply, and/or through a waste-heat boiler.

A. W. Hothersall.

Apparatus for determining temperatures by carbon dioxide content of combustion gases. F. D. Potter, Assr. to Combustion Control Co. (U.S.P. 1,577,853, 23.3.26. Appl., 24.3.19).—The furnace gases pass through a short-circuit passage in the furnace wall to a carbon dioxide recorder. The usual record sheet is replaced by a chart which directly indicates temperatures and corresponding carbon dioxide contents of the gases. Provided the air admitted to the furnace is equal to, or in excess of, that required for complete combustion, the percentage of carbon dioxide in the gases varies inversely as the quantity of air supplied and directly as the temperature reached.

R. B. Clarke.

Quantitative determination of selected gases in atmospheres. L. B. Atkinson (E.P. 248,807, 13.10.24).—Gases are estimated quantitatively by the use of a standardised test paper impregnated with a reagent and indicator. The paper is prepared by being dipped into a solution of the reagent of suitable concentration, leaving part unmoistened, and so obtaining an approximately uniform concentration

of reagent over the moistened area. A paper impregnated with alkali and phenolphthalein may be used for estimating small amounts of carbon dioxide in air by determining the time taken for the colour change to occur, the paper having been calibrated in standard gas mixtures. A. B. MANNING.

Quantitative determination of gases [carbon monoxide]. S. H. KATZ (U.S.P. 1,578,666, 30.3.26. Appl., 12.3.25).—Carbon monoxide is determined in gas mixtures by passing the latter over hopcalite (cf. Lamb, Bray, and Frazer, B., 1920, 424 A) and measuring the rise in temperature produced. Before entering the constant-temperature chamber containing the catalytic cell, the gases are filtered from suspended material which might poison the catalyst, and are preheated to the temperature of the chamber. The rise in temperature is measured electrically. A. B. MANNING.

Scrubber for separating oil and other liquid particles from gas. SMITH SEPARATOR Co., Assees. of M. F. WATERS (E.P. 234,436, 17.11.24. Conv., 23.5.24).—The scrubber consists of a vertical cylindrical vessel, the "wet" gas entering about half-way up, and being deflected downwards by a baffle. The gas on rising to the upper part of the vessel encounters a conical baffle (apex downwards) filling the whole cross section of the vessel; staggered ports are provided in this baffle, permitting the passage of the gas, the ports each having the shape of a truncated cone (apex upwards). At a still higher level is a non-perforated conical baffle (apex downwards), fixed so as to leave an annular space between the edge of the baffle and the sides of the vessel. By this system of baffles the gas is progressively dried, and passes away from the top of the vessel. The separated oil is conveyed by drain pipes to the lower part, and is drawn off as required. W. N. HOYTE.

Purifying coal or other gases. J. N. REESON and W. L. MOSS (E.P. 249,312, 11.3.25).—Hydrogen sulphide, carbon disulphide, and thiohydrocarbons may be removed from coal gas or other gases by washing the gas in a solution prepared by adding to a 2% solution of ferrous sulphate (or chloride), 1—5% of an organic substance which will prevent the precipitation of iron hydroxide when the solution is neutralised, *e.g.*, salicylic acid, *p*-hydroxybenzoic acid, phthalic acid, polyhydric alcohols and their derivatives including the glycols and glycerol, and citric acid. The solution is then neutralised with soda ash and oxidised by aeration. It can be regenerated when necessary by aerating with air or oxygen at a suitable temperature and under pressure, and afterwards neutralising with alkali and adding iron salts to replace loss. The sulphur formed during the regeneration process can be removed and the accumulated sulphur compounds can be ultimately recovered as sodium thiosulphate and sulphate. A. W. HOTHERSALL.

Purifying gas-house liquor. E. W. KEITH, Assr. to GEN. METALS RECOVERY Co. (U.S.P. 1,573,169, 16.2.26. Appl., 30.9.24).—Gas-house liquor is made suitable for hydro-metallurgical use by treating it with zinc oxide in quantity equivalent to the ammonium sulphide present. The precipitated zinc sulphide is removed and calcined to give the oxide for further use. R. B. CLARKE.

Removal of naphthalene and analogous hydrocarbons from fuel gases. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,578,687, 30.3.26. Appl., 26.1.25).—In a two-stage process for removing naphthalene the gas is washed first with oil which is continuously circulated in contact with the gas, and then with fresh oil. S. PEXTON.

Utilisation of liquid slag from gas producers and pulverised fuel furnaces. G. POLYSIUS EISENGIESSEREI & MASCHINENFABR. (G.P. 423,793, 5.5.22. Addn. to 421,427).—The process described in the chief patent (B., 1926, 363), is carried out in rotating kilns, thereby obtaining a more even distribution of heat and a better admixture of the slag and the added constituents. B. W. CLARKE.

[Motor] fuel. A. WOOSNAM. From CANADIAN ELECTRO PRODUCTS Co. (E.P. 249,348, 29.4.25).—Between 2% and 20% of an aldehyde (in particular paraldehyde) saturated with acetylene, is dissolved in motor spirit. It is claimed that the deposition of carbon in the cylinder of the engine is reduced to negligible quantities. About 12 vols. of acetylene are dissolved in 1 vol. of the aldehyde. The heavier the motor spirit the more aldehyde is required; ordinary grades of petrol require 3·5—10%. W. N. HOYTE.

[Motor] fuel. T. MIDGELEY, jun., Assr. to GEN. MOTORS CORP. (U.S.P. 1,578,201, 23.3.26. Appl., 28.2.20).—The fuel consists of 50% of commercial ethyl alcohol and 50% of a cracked gasoline, the latter containing 84% of saturated hydrocarbons and 16% of unsaturated (olefines). W. N. HOYTE.

Agent for raising the calorific power of motor fuels. D. COSTAGUTA (F.P. 599,271, 9.6.25).—The agent consists of a nitrophenol, *e.g.*, trinitrophenol, one or more ethers, and one or more liquid hydrocarbons. It is added to the motor fuel in the proportion of 4·5%. B. FULLMAN.

Decomposing oils. C. M. ALEXANDER (U.S.P. 1,573,532, 16.2.26. Appl., 9.10.19).—A heavy oil is maintained at a temperature of 370—425° and preheated light oil to be cracked is introduced in a spray below its surface so that cracking takes place before vaporisation. The vapours from the cracking still are fractionated and any uncracked oil is returned with fresh light oil. The heavy oil is continuously circulated from the still through a chamber in which any carbon it contains is removed. T. S. WHEELER.

Apparatus for treating [cracking] oil. F. C. VAN DE WATER and F. R. SUNDERMAN, Assrs. to PETROLEUM LABORATORIES, INC. (U.S.P. 1,575,663, 9.3.26. Appl., 16.1.23).—A cracking still is provided

with a dome, and a supplementary chamber within the dome has a series of diametrically opposed valved openings, one set of which is connected with a supply of gas, *e.g.*, hydrogen, and another set with the still body. H. MOORE.

Apparatus for distilling hydrocarbons. F. E. WELLMAN, Assr. to KANSAS CITY GASOLINE Co. (U.S.P. 1,576,564, 16.3.26. Appl., 3.6.22).—A cracking still is connected with a fractionator, and this with a supplementary still connected with a second fractionator. Both fractionators are connected with a condenser. A return pipe leads from the supplementary still to the cracking still. A feed pipe supplies fresh oil through the first fractionator and supplementary still. H. MOORE.

Apparatus for refining [cracking hydrocarbon] oils. W. T. HANCOCK and M. BOYLE (U.S.P. 1,576,742, 16.3.26. Appl., 8.10.23).—A still for the conversion of heavy hydrocarbon oils into lighter oils has perforated pipes running longitudinally, their ends being supported by sheets spaced from the ends of the still to provide steam spaces. There are steam inlets at the ends of the still and a separator chamber connected to its upper portion, a condenser being connected with the top of this chamber. H. MOORE.

Process for separating hydrocarbons. W. C. AVERILL, JUN. (U.S.P. 1,577,440, 23.3.26. Appl., 10.12.23).—Hydrocarbon oil containing water is vaporised and the water vapour is condensed completely together with part of the oil. The remaining oil vapours are scrubbed with oil of high boiling point obtained from a previous distillation and are then subjected to further condensation. The scrubbing oil is that obtained in the first partial condensation. W. N. HOYTE.

Continuous catalytic distillation [of hydrocarbons]. G. L. PRICHARD and H. HENDERSON, Assrs. to GULF REFINING Co. (U.S.P. 1,577,871, 23.3.26. Appl., 14.2.22).—Hydrocarbons of high boiling point are distilled in the presence of aluminium chloride as catalyst. From the vapours the fractions of higher boiling point and the catalyst are first separated, and the vapours then pass on for final condensation. The mixture of catalyst and oil of high boiling point is returned to a second batch of cracking stock and catalyst, which in its turn is distilled. W. N. HOYTE.

Cracking hydrocarbon oil. E. W. ISOM, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,578,035, 23.3.26. Appl., 19.6.25).—Oil is passed through a pipe still heated by furnace gases, and is brought up to cracking temperature. The cracked oil vapours pass through a reflux condenser and the reflux is returned to the main flow of oil. Fresh oil is added as required. The heating gases are circulated repeatedly over the pipes, fresh gases being added as required. W. N. HOYTE.

Distilling oils with volatile catalysts. A. McD. MCAFEE, Assr. to GULF REFINING Co. (U.S.P. 1,578,049, 23.3.26. Appl., 20.1.22).—A mixture of the oil with a metal halide as catalyst is distilled, and the resulting vapours of products of lower boiling point are condensed after having been brought into intimate contact with a petroleum hydrocarbon. W. N. HOYTE.

Distilling [petroleum oil] with aluminium chloride. A. McD. MCAFEE, Assr. to GULF REFINING Co. (U.S.P. 1,578,050, 23.3.26. Appl., 20.1.22).—The vapours resulting from the distillation of the oil with aluminium chloride as catalyst are partially condensed to separate the catalyst and kerosene, and then passed to a final condenser. The catalyst is separated by settling from the kerosene, and is returned to the still. W. N. HOYTE.

Process for improving oils. A. McD. MCAFEE, Assr. to GULF REFINING Co. (U.S.P. 1,578,051, 23.3.26. Appl., 20.1.22).—A flow of oil is maintained through a series of stills operated in succession and is distilled in presence of aluminium chloride as catalyst. The volatilised chloride is condensed, collected, and returned towards the first still of the series. W. N. HOYTE.

Removing aluminium chloride residues from [oil] stills. A. McD. MCAFEE, Assr. to GULF REFINING Co. (U.S.P. 1,578,053, 23.3.26. Appl., 13.12.23).—The caked mass of chloride is disintegrated by wetting with water, is then flushed out of the still with water. W. N. HOYTE.

Lubricating greases. RÜTGERSWERKE-A.-G., and H. TEICHMANN (G.P., 382,184, 22.7.20).—The lubricants contain resinous matters obtained by the action of high-boiling coal-tar oils on coal, wood, or other substances of organic structure; they are sometimes admixed with other lubricants. They have a high drop-point and are insensitive to temperature changes. B. FULLMAN.

Combustion of solid fuel. M. DE CONINCK (U.S.P. 1,580,656, 13.4.26. Appl., 24.1.24).—See E.P. 210,419; B., 1924, 932.

Eliminating the sulphuretted hydrogen in fuel gas. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,578,560, 30.3.26. Appl., 6.6.24).—See E.P. 241,248; B., 1926, 5.

Removing aluminium chloride residues from vessels used for the catalytic treatment of petroleum hydrocarbons. A. J. H. HADDAN. From GULF REFINING Co. (E.P. 250,102, 11.9.25).—See U.S.P. 1,578,053; preceding.

Dehydrating [oil] emulsions. W. CAMERON, Assr. to SIMPLEX REFINING Co. (U.S.P. 1,580,956, 13.4.26. Appl., 15.11.20).—See E.P. 173,644; B., 1922, 131 A.

Maintaining solid [coal] particles uniformly suspended in liquids, more especially liquid fuels. G. E. HEYL (E.P. 250,490, 17.11.25).

Refining of hydrocarbons (E.P. 249,309).—See III.

Extracting hydrogen from industrial gases (E.P. 242,583).—See VII.

Impregnating wood (E.P. 244,449).—See IX.

Distillation of hydrocarbons etc. (G.P. 424,678).—See XII.

III.—TAR AND TAR PRODUCTS.

Benzol recovery by vacuum method. GAREIS.—See II.

PATENTS.

Refining of hydrocarbons. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 249,309, 9.3.25).—Tar from ordinary or low-temperature carbonisation of coal etc., petrol, cracked oil, or the like is distilled over caustic soda or lime using non-superheated steam. The distillate may be further refined by passing the vapours together with hydrogen over finely-divided nickel, copper, iron, etc. at 100—110°. W. N. HOYTE.

Separation of constituents of low-temperature coal-tar without distillation. ZECHE M. STINNES, and A. WEINDEL (G.P. 420,394, 5.9.23).—The tar is washed several times on the counter-current principle with sodium hydroxide of lower strength than 5%. The acid constituents are removed, and the neutral oil is separated unchanged in properties (*e.g.*, viscosity). For example, the tar is intimately stirred with 3% sodium hydroxide at normal or slightly raised temperature, and left for a short time in a separator. At first a large part of the sodium hydroxide is absorbed, and then separation occurs. Four washings are usually sufficient. The phenols are precipitated from the alkaline liquid floating on the neutral oil by acid treatment. B. FULLMAN.

Lubricating greases (G.P. 382,184).—See II.

Distillation of hydrocarbons etc. (G.P. 424,678).—See XII.

IV.—DYESTUFFS AND INTERMEDIATES.

Use of chlorine in the organic chemical industry. J. T. CONROY (Trans. Amer. Electrochem. Soc., 1926, 49, 47—64. Advance copy).

Synthetic colouring matters used in lake making. ROWE.—See XIII.

PATENTS.

Azo dyestuffs. BADISCHE ANILIN- & SODA-FABR., Assees. of F. GÜNTHER (U.S.P. 1,573,605, 16.2.26. Appl., 13.7.25).—Compounds containing the grouping R·N:N·Ar(*p*)·X(*p*)·Ar·N:N·R in which Ar stands for an aryl group, R is a salicylic acid derivative, and X is an azo, azoxy, or ·NH·CO·NH· group, and also sulphonic acid groups, dye animal, vegetable, or mixed fibres very fast shades. To prepare them a *p*-nitroarylaminesulphonic acid is diazotised and coupled with salicylic acid and the product reduced to the azo- or azoxy-derivative to yield a compound which gives directly orange to red-brown shades on fibres. Alternatively the azo dyes from *p*-nitroarylamines and salicylic acid are sulphonated and reduced to the corresponding azo- or azoxy-compounds, or sulphonation is performed after reduction. For the carbamide derivatives the unsulphonated dyestuffs from diazotised diaminodiarylcarbamides and 2 mols. of salicylic acid or its homologues or derivatives are sulphonated or a sulphonated diaminodiarylcarbamide is diazotised and coupled with 2 mols. of salicylic acid, or a *p*-nitroarylaminesulphonic acid is diazotised and coupled with salicylic acid, the nitro-groups are reduced to amino-groups, and the product is treated with phosgene. The carbamide derivatives give light yellow shades. For example *p*-nitroaniline-*o*-sulphonic acid is diazotised and coupled with salicylic acid and the azo-compound is reduced with sodium sulphide to give an azo- or azoxy-derivative which dyes cotton directly. Treated with chromium salts a chromium compound of greatly increased fastness yielding copper shades on cotton is obtained. T. S. WHEELER.

Azo dye. FARBENFABR. VORM. F. BAYER & Co., Assees. of J. HALLER (U.S.P. 1,576,322, 9.3.26. Appl., 7.11.25).—A diazotised *as.-m*-xylidine (1:3-dimethyl-4-aminobenzene) or its 6-nitro-derivative is coupled with a 2:3-hydroxynaphthoic acid *as.-m*-xylidide to yield azo dyes which give fuller and clearer shades on vegetable fibres than do the analogous compounds obtained with 2:3-hydroxynaphthoic acid anilide. The shades are very fast to light and kier-boiling. For example, cotton is treated first with 2:3-hydroxynaphthoic acid *as.-m*-xylidide in caustic soda solution and Turkey-red oil and then with diazotised *as.-m*-xylidine, when a bluish-red shade is developed. The diazotised 6-nitroxylidine coupled with the 6-nitro-xylidide gives yellow-red shades. T. S. WHEELER.

Substantive green azo dyes. A.-G. FÜR ANILIN-FABR., Assees. of W. LANGE and L. NEUMANN (G.P. 419,585, 22.2.24).—The dyes are obtained by treating in aqueous solution with phosgene a mixture of equivalent quantities of a blue trisazo dye of the type $A{\cdot}N_2{\cdot}M^{1}{\cdot}N_2{\cdot}M^{11}{\cdot}N_2{\cdot}M^{111}$ (where A is an aromatic residue, M^1 and M^{11} are aromatic residues having no hydroxyl groups, and M^{111} is a hydroxy-amino-residue of the naphthalene series giving no ring formation with phosgene) and a yellow aminoazo-compound. A dye which produces green shades fast to light on cotton is thus obtained from 1-aminobenzene-2 : 5-disulphonic acid-azo-1-aminonaphthalene-7-sulphonic acid-azo-1-aminonaphthalene-azo-2-amino-5-hydroxynaphthalene-7-sulphonic acid and 1-aminobenzene-3-carboxylic acid-azo-2-amino-1-methoxybenzene. L. M. CLARK.

Manufacture of new azo dyes and intermediate products. A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 248,946, 15.4.25).—New derivatives of the arylides of 2 : 3-hydroxynaphthoic acid of the general formula,

$C_{10}H_6(OH){\cdot}CO{\cdot}NH{\cdot}aryl{\cdot}X{\cdot}aryl{\cdot}NH{\cdot}OC{\cdot}C_{10}H_6(OH)$

(where X=an azo or azoxy group) are obtained either by condensing the chloride of 2 : 3-hydroxynaphthoic acid or an *O*-acyl derivative with an aromatic diamino-azoxy or diamino-azo compound

(afterwards removing the acyl group if present), or by treating a nitroarylide of 2 : 3-hydroxynaphthoic acid with reducing agents, such as dextrose, arsenious acid, zinc dust, or formaldehyde in alkaline solution. These compounds couple with diazo compounds to form azo dyes. The above arylide derivatives have an affinity for vegetable fibre equal to that of direct cotton dyes. By treating tne fibre with these derivatives and then with a diazo compound dyeings specially fast to rubbing are obtained, the intensity of which depends on the introduction of a further chromophore into the molecule on the side of the arylide of 2 : 3-hydroxynaphthoic acid. The new dyes are evenly absorbed from a hot bath and are specially adapted for the dyeing of heavy goods. For example, 52 pts. (by weight) of the chloride of 2 : 3-hydroxynaphthoic acid and 21 pts. of *p* : *p'*-diaminoazobenzene, each dissolved in acetone, are stirred together for 1 hr. and the mixture is made feebly alkaline with sodium hydroxide. The precipitated *di*-2 : 3-*hydroxynaphthoyl*-*p* : *p'*-*diaminoazobenzene*, a yellowish-brown powder, has m.p. above 360°. *Di*-2-3-*hydroxynaphthoyl*-*m* : *m'*-*diaminoazoxybenzene*, a brownish crystalline powder, decomp. above 285° without melting, is obtained by dissolving 30·8 pts. of the *m*-nitroanilide of 2 : 3-hydroxynaphthoic acid in 22 pts. of aqueous sodium hydroxide (*d* 1·383) and 500 pts. of water at 65° and reducing with a solution of 12 pts. of dextrose in 40 pts. of water at 60—70° for 1—2 hrs. 29·8 pts. of the azoxy-compound, so obtained, are dissolved in 12 pts. of aqueous sodium hydroxide (*d* 1·383), together with some Turkey-red oil and sufficient sodium acetate or carbonate for neutralising the diazo-solution, and treated with 12·9 pts. of diazotised *p*-chloroaniline. The dye separates as bluish-red flakes. As an example of development on the fibre, 0·4 pt. of the azoxy-compound obtained by reduction of the *m*-nitroanilide of 2 : 3-hydroxynaphthoic acid in 0·8 pt. of aqueous sodium hydroxide (*d* 1·383) is mixed with a little Turkey-red oil and brought up to 200 pts. with hot water. 10 pts. of cotton are introduced at the boil and dyed for 1 hr. while cooling. Development with diazotised *m*-nitro-*p*-toluidine gives a clear bluish Turkey-red shade of good fastness to rubbing and washing. The tints (chiefly yellowish-red to brown) of about 80 dyes are given. A. COULTHARD.

[Perylene] vat dyes. H. SCHÖPFER, Assr. to F. BENSA (U.S.P. 1,574,116, 23.2.26. Appl., 9.4.25).—By heating dinitro- or tetranitro-perylene with 5 pts. of anhydrous aluminium chloride for 4 hrs. at 160—200°, condensed nitroperylenes are obtained. That from the dinitro-compound dyes cotton in the vat fast olive-brown shades; that from the tetranitro-derivative gives olive-green shades.
T. S. WHEELER.

Manufacture of ω-aminoalkylaminonaphthalene compounds and substitution products thereof. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER & Co. (E.P. 249,717, 29.6.25).—The compounds are prepared by treating naphtholcarboxylic acids containing the hydroxyl and carboxyl groups in the *ortho* position to each other, with aliphatic diamines, in the presence of sodium bisulphite. For example, a mixture of 40 pts. of ethylenediamine hydrate, 300—400 pts. of sodium bisulphite solution, *d* 1·357, and a solution of 38 pts. of 2-naphthol-3-carboxylic acid in the requisite quantity of sodium hydroxide solution is heated to 90—100° with stirring until no more carbon dioxide is evolved. After cooling, the pale yellow crystalline precipitate is removed, washed with salt solution and water, stirred with hot, dilute sodium hydroxide solution to remove residual β-naphthol, and again filtered and washed with water. The residue consists of *β-naphthyl-β-aminoethylamine*. The preparation of *β-naphthyl-δ-aminobutylamine* and *β-naphthyl-β-aminoethylaminoethylamine* is also described.
L. A. COLES.

Dyestuff intermediates. M. T. BOGERT (U.S.P. 1,574,337, 23.2.26. Appl., 15.10.24; *cf.* B., 1926, 354).—2:4-Dinitrodiaryl ethers or 2:4-nitroaminodiaryl ethers are readily reduced to the corresponding 2:4-diaminodiaryl ethers by treatment with iron, water, and a little hydrochloric acid at 100°. These compounds yield useful dyestuffs by reactions similar to those used to obtain analogous dyestuffs from *m*-phenylenediamine. 2:4-*Diaminodiphenyl ether* has m.p. 67°; *diacetyl* derivative, m.p. 171° *hydrochloride* softens at 190°, m.p. about 210°.
T. S. WHEELER.

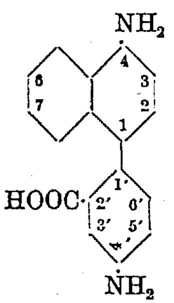
Hydrogenation of naphthalene. "PRAX" CHEM. VERSUCHS- U. VERWERTUNGS-G.M.B.H. (F.P. 533,033, 30.3.21. Conv., 5.10.16).—Naphthalene is mixed with nickel carbonate and treated with hydrogen at 200—280°. A colloidal solution of nickel results, followed by reduction. B. FULLMAN.

Separation of *o*- and *p*-chloronitrobenzene. VEREIN FÜR CHEM. U. METALLURGISCHE PRODUKTION (G.P. 420,129, 29.4.22).—The eutectic mixture of 66·9% of *o*- and 33·1% of *p*-chloronitrobenzene, m.p. 14·65°, is melted with *p*-dichlorobenzene, and the melt cooled to 11°. A mixture of *p*-dichlorobenzene with the greater part of the *p*-chloronitrobenzene crystallises out, and the two compounds are separated by steam distillation. The liquid residue from the eutectic mixture is steam-distilled, and cooled to 15°, when *o*-chloronitrobenzene separates, and may be freed from residual eutectic mixture by centrifuging. B. FULLMAN.

Preparation of phenols. BADISCHE ANILIN- & SODA-FABR., Assees. of O. SCHMIDT and K. SEYDEL (G.P. 420,393, 28.7.23).—Phenol homologues may be converted into lower members by treating them at high temperatures with hydrogen in the presence of activated catalysts (*e.g.*, Ni, Cu, Ag, Pt, using as activators the substances mentioned in G.P. 307,580 and 408,811; cf. B., 1925, 475) under such conditions that ring-hydrogenation does not occur. For example, if *o*-cresol vapour be led at 400° over a catalyst formed by reduction of nickel chromate on pumice stone, a good yield of phenol is obtained, methane being evolved. With a similar catalyst the vapour of mixed xylenols yields a mixture of phenol and cresol. B. FULLMAN.

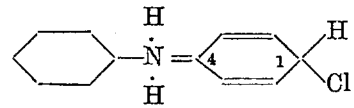
Oxidation of organic substances and the preparation of salt mixtures containing chromate and dichromate. C. F. BOEHRINGER & SOEHNE G.M.B.H. (G.P. 420,444, 24.10.23).—Oxidation is carried out with alkali chromate or dichromate in nitric acid solution, the exhausted liquor concentrated with recovery of the nitric acid which distils, and the residue fused, the resulting nitrous fumes being converted into nitric acid; or chromium nitrate or products containing it are heated to fusion with alkali nitrates, the nitrous fumes being regenerated. For example, anthracene suspended in 10% nitric acid is quantitatively oxidised to anthraquinone by addition of solid sodium dichromate at 75—80°. The residual liquor is treated with an excess of equal parts of sodium and potassium nitrates, evaporated, and the residual solid fused at 280—300°. Aniline is oxidised in 12% nitric acid by addition of sodium dichromate solution, at 5—10°. After extraction of the quinone, the liquor is evaporated, fusion of the residue yielding sodium dichromate. B. FULLMAN.

Production of benzanthrone derivatives. L. CASSELLA UND CO., Assees. of G. KALISCHER, R. MÜLLER, and F. FRISTER (U.S.P. 1,565,229, 8.12.25. Appl., 19.3.25).—α-Naphthaleneazobenzene-*m*-carboxylic acids are reduced in acid solution and the diamino-substituted phenylnaphthalenes thus obtained, of the annexed formula in which the C-atoms, 2, 3, 5, 6, 7, 3′, 5′, 6′ may be substituted by Me, halogens, or alkoxy-groups, are condensed to the corresponding diaminobenzanthrones by treatment with acid-condensing agents. For example, the sodium salt of *α-naphthaleneazobenzene-m-carboxylic acid* (the free acid, m.p. 207°, is obtained by boiling the diazo-compound of the dyestuff from *m*-diazobenzoic acid and α-naphthylamine with alcohol) is reduced with stannous chloride and the resulting dihydrochloride of 1-(4-*amino*-2-*carboxyphenyl*)-4-*aminonaphthalene* is condensed with chlorosulphonic acid to give 4:4′-*diaminobenzanthrone*, m.p. 225°. Diazotised *m*-amino-*p*-methylbenzenecarboxylic acid and α-naphthylamine when coupled and the product diazotised and boiled with alcohol give the corresponding *α-naphthaleneazo-6′-methyl-3′-benzenecarboxylic acid* m.p. 226°, and this treated as above gives the corresponding 4:4′-*diamino-5′-methylbenzanthrone.* T. S. WHEELER.

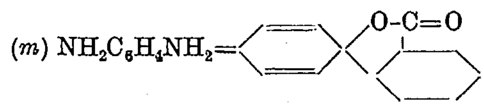


Preparation of 5-hydroxy-*N*-methyloxindole [and *p*-hydroxyphenylmethylglycine]. E. MERCK, CHEM. FABR., Assees. of A. DÜTZMANN (G.P. 421,386, 23.8.24).—*N*-Methyl-*p*-aminophenol sulphate is treated in aqueous solution with 25% ammonia and a little aqueous sulphur dioxide. The free base is washed with aqueous sulphur dioxide, boiled for several hours with glyoxal sodium sulphite, filtered, and concentrated to crystallisation. The product is recrystallised from 20% sodium acetate solution, and 5-*hydroxy*-N-*methyloxindole*, m.p. 186—187°, obtained from the purified product (its sodium sulphite) by treatment with hydrochloric acid. The filtrate from the sodium sulphite derivative yields, on treatment with acetic acid, N-p-*hydroxyphenylmethylglycine*. B. FULLMAN.

Dyestuffs. W. P. BATES (U.S.P. 1,572,417, 9.2.26. Appl., 25.8.24).—By the interaction of an aromatic diazonium chloride with an aromatic amine or diamine in presence of water at about 90°, a quinonoid compound with the formula



is obtained. Compounds of this type are either dyes for feathers, furs, hair, wool, and the like, or may be used as intermediates for the manufacture of dyes for such purposes. The hydrogen and chlorine in position 1 are readily replaced by various atoms or groups, and the NH_2 group in 4 is also reactive. The product from benzenediazonium chloride and *m*-phenylenediamine dyes furs, feathers, and hair brown. The substitution of the chlorine atom by methoxyl by treatment with sodium methoxide improves the shade. Sodium salicylate reacts with removal of sodium chloride and formation of an ester derivative which when treated with fused zinc chloride loses water and gives a compound with the formula



which gives orange shades on wool in the cold. Replacement of chlorine by a *p*-hydroxyphenylglycine residue yields a product which gives yellow-orange shades, and substitution by carboxyl through the nitrile yields a product giving deep yellow shades. Sulphonation gives products which dye silk from an acid bath rich golden brown to yellow shades. T. S. WHEELER.

Preparation of quinol. A. ERLACH (Austr. P. 98,418, 21.4.23).—Benzoquinone, prepared by the oxidation of aniline by means of chromium trioxide in sulphuric acid solution, is separated from the acid liquor by distillation *in vacuo* in a current of steam in the presence of boric acid, and is subsequently reduced to quinol by treatment with sulphur dioxide. A yield of quinol of about 65—80% of the weight of the aniline is obtained. L. A. COLES.

Process of manufacturing [benzanthrone] vat dyes. BADISCHE ANILIN- & SODA-FABR., Assees. of A. LÜTTRINGHAUS, H. NERESHEIMER, and H. EMMER (U.S.P. 1,580,062, 6.4.26. Appl., 11.11.24).—See F.P. 589,323; B., 1926, 148.

o-Benzylphenol. FARBENFABR. VORM. F. BAYER & Co., Assees. of W. KROPP, W. SCHRANZ, and W. SCHULEMANN (U.S.P. 1,580,053, 6.4.26. Appl., 1.2.24).—See G.P. 406,532; B., 1925, 474.

Diacylacetyldiamino-compounds of the aromatic series. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. ZITSCHER and R. SCHMITT (U.S.P. 1,580,709, 13.4.26. Appl., 15.5.23).—See E.P. 211,772; B., 1924, 413.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

[Textile] piece [goods] carbonising. M. L. WILSON (J. Soc. Dyers and Col., 1926, **42**, 109—110).—Burrs and vegetable impurities are satisfactorily removed from woollen fabrics by carbonising for 20—30 min. at 71° with 7·5% sulphuric acid, and the resulting fabric can be dyed evenly provided that the fabric before carbonising is thoroughly freed from grease and soap and not allowed to lie more than 20—30 min. after removal of the excess acid by mangling or hydro-extraction.

A. J. HALL.

[Pulverulent cellulose and chlorinated hydrocellulose.] C. DE NEYMAN (Chim. et. Ind., 1926, **15**, 349—352).—Powdered cellulose is prepared by treating paper with sulphuric or hydrochloric acid or with ferric chloride and disintegrating the mass mechanically. The quantity of acid used and the temperature govern the nature of the product. Much acid and high temperature result in a coloured powder. By treating cellulose with chlorine gas a chlorinated hydrocellulose is produced, characterised by the ease with which it can be acetylated by means of a mixture of acetic acid and acetic anhydride. In this process acetic acid—used merely as a diluent—may be replaced by other neutral liquids in which cellulose acetate is insoluble. Benzene, petroleum spirit, or paraffin oil may be used for the purpose but the proportion of acetic anhydride must be slightly increased. A better grade of cellulose acetate is obtained by the use of cotton and the reaction is accelerated by the addition of small quantities of sulphuric acid. Previous moistening of the cellulose with acetic acid aids the admixture with the acetylating agents. A. G. POLLARD.

Chemical processes in the ripening of viscose. LEUCHS (Kunstseide, 1925, **7**, 286; Chem. Zentr., 1926, I., 2415).—Sodium hydroxide and carbon disulphide react according to the equation $5CS_2 + 12NaOH = Na_2S + 3CS(SNa)_2 + 2Na_2CO_3 + 6H_2O$. By the action of acid the 1 mol. of sodium sulphide yields 1 mol. of hydrogen sulphide, and the 3 mols. of sodium trithiocarbonate yield 3 mols. of hydrogen sulphide and of carbon disulphide. If by treating viscose with acid a % of hydrogen sulphide and b % of carbon disulphide are obtained, $\frac{1}{4}a$ arises from sodium sulphide and $\frac{3}{4}a$ from sodium trithiocarbonate, which latter also gives an equal molecular amount of carbon disulphide which was combined with cellulose. During the ripening of viscose the xanthate is slowly hydrolysed and the dithiocarbonic acid residues thus formed react with sodium hydroxide. The differences in the amounts of carbon disulphide liberated by acid at different stages of ripening are only 2/5 of the amounts produced by the decomposition of the xanthate. The differences are so small that an illusory constancy of the ratio of carbon disulphide to cellulose is obtained.

A. GEAKE.

Absorption of sodium hydroxide from its solutions and from "black liquor" by sulphate-cellulose. C. KULLGREN (Papier-Fabr., 1926, **24**, 153—155, 185—189, 206—208).—The absorption of sodium hydroxide from its solutions by sulphate-cellulose, as kraft cellulose and as bleachable pulp, was determined by the indirect method. 2 g. of cellulose were shaken with 100 c.c. of the solution and 50 c.c. were then titrated with acid. When the concentration of sodium hydroxide was 3·5 g. per 100 c.c. absorption was complete in 2—60 min., according to the conditions of shaking, and it diminished with rising temperature between 18° and 50°. At 75° acidic substances were formed by oxidation of the cellulose by atmospheric oxygen. With increasing concentration of sodium hydroxide from 0 to 7% the absorption curve was bent first towards the concentration axis and then away from it. It is probable that a strongly hydrolysed compound of sodium hydroxide and cellulose is formed. The absorption is reversible, and the equilibrium may be reached from either side. No absorption of sodium chloride or carbonate could be detected but the addition of the former to sodium hydroxide markedly increases the absorption of this latter. The absorption of sodium hydroxide from solutions of sodium sulphide, which is nearly completely hydrolysed in dilute solution, and from black liquor from sulphate-cellulose manufacture was determined by comparing the change in electrical conductivity when kraft cellulose was added with that produced by the addition of small amounts of sodium hydroxide. The method is not very accurate on account of the large temperature coefficient of the conductivity. The absorption in both cases is greater than from pure sodium hydroxide solutions of the same alkalinity, and this is ascribed to the presence of other sodium salts. It is computed that the loss due to absorption from black liquor amounts to 4—8 kg. of sodium sulphate per ton of cellulose. A. GEAKE.

Determination of lignin in wood and wood cellulose. W. J. MÜLLER and W. HERRMANN (Papier-Fabr., 1926, **24**, 185).—After dissolution of the cellulose by any of the known methods, the solution, containing the lignin in suspension, is filtered through a layer of finely-divided naphthalene in a glass or porcelain filter-crucible. To prepare the filter a suitable volume (*e.g.*, 10—20 c.c.) of a 5% solution of naphthalene in alcohol is poured into twice its volume of water, and the paste thus obtained introduced into the filter, pressed down, and washed. After use the naphthalene is sublimed over a water-bath and the lignin is thus obtained pure and loose.

A. GEAKE.

PATENTS.

Manufacture of cellulosic material for paper, textiles, and the like. A. L. BURLIN, S. LEICESTER, and L. B. HOLMAN (E.P. 248,118, 10.12.24).—Cellulosic material containing lignone, or of a silicious character, is treated with bleaching powder (0·5 lb. per gall.) and sodium bicarbonate (2—4 lb. per 100 lb. of material) at a relatively low temperature, *e.g.*, 28°. When the cellulose is lignified it may have a preliminary treatment with an organic acid, *e.g.*, benzoic acid. A final treatment with a fixing agent, *e.g.*, alum, may be given to prevent subsequent discoloration. The process may be applied to mechanical wood pulp, or to the manufacture of paper pulp from waste jute material. A. GEAKE.

Manufacture of cellulosic products [esters]. H. DREYFUS (E.P. 249,173, 25.9.24).—Wood pulp or similar material is converted into cellulose sufficiently pure for esterification, by repeated or prolonged treatment with hot or boiling solutions containing ¼—3% (preferably not more than 2%) of an alkali hydroxide, or with solutions at the ordinary or at slightly raised temperatures, containing about 5—10% of an alkali hydroxide, followed by boiling with dilute or, preferably, with glacial acetic acid, or with other lower aliphatic acids. The alkali hydroxides may be replaced by other basic materials, such as sodium carbonate, potassium carbonate, barium hydroxide, sodium zincate, sodium aluminate, ammonia, or organic bases, in which case higher concentrations are used, and, if necessary, the treatment is effected under increased pressure. In treating mechanical wood pulp or other material from which lignocellulose, resins, and similar constituents have not been removed in a preliminary process, the basic material is used in sufficient quantity to ensure elimination of these constituents and to bring the cellulose into the desired condition. L. A. COLES.

Manufacture of filaments and films from viscose. W. MENDEL (U.S.P. 1,576,529–31, 16.3.26. Appl., 29.4.25).—Undesirable sulphur-containing constituents are removed from cellulose products by treatment with trisodium phosphate; thus viscose is so treated after precipitating (A) in an acid bath, (B) in an aqueous solution containing sodium hydrogen sulphite, or (C) by heat. A. GEAKE.

[Lamp] wicks. F. D. MILES, and NOBEL'S EXPLOSIVES CO., LTD. (E.P. 249,571, 26.9.24).—Wick is tendered by heating with hydrochloric or nitric acid and the surface then reinforced by treatment with a nitrocellulose solution. Wick so treated is easily cut level and is not distorted in use. A. GEAKE.

Treating viscose silk in a vacuum. A. KÄMPF (U.S.P. 1,579,711, 6.4.26. Appl., 26.3.25).—See E.P. 232,200; B., 1925, 914.

Artificial horn (E.P. 230,025).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Cotton: affinity of derivatives of dehydrothiotoluidine and primuline. P. RUGGLI and S. M. PESTALOZZI (Helv. Chim. Acta, 1926, 9, 364—378).—A cotton skein weighing 5 g. was boiled for 1 hr. with 0·15 g. of dye and 100 c.c. of distilled water and the amount of dye absorbed from solution was determined. The skein was then rinsed and treated with 100 c.c. of boiling water and the dye stripped out estimated after 24 hrs. The difference between these two values, expressed in % of the amount of dye originally taken is proposed as an empirical measure of the affinity of a dye for cotton. The affinity is estimated for a range of substances in the dehydrothiotoluidine and primuline classes. The affinity of the sodium salt of dehydrothiotoluidine-3-sulphonic acid is almost negligible but it can be developed by introduction into the molecule of suitable substituents such as nitro- or amino-groups. The affinity is markedly increased by introduction of a second thiazole nucleus to give the corresponding primulinesulphonic acid. In the azo-derivatives of these compounds, however, the lower homologue shows, in the cases considered, the higher affinity, although the primuline dyes are less readily stripped from the cotton. The choice of coupling components in the preparation of these derivatives has considerable influence on the affinity. The dehydrothiotoluidine and primuline azo-dyes, as a class, show small affinity compared with that of the bisazobenzidine dyes. When the dyeing process is carried out in the presence of sodium sulphate, an increased affinity is obtained, notwithstanding the greater ease with which the dye is stripped from the cotton afterwards. The degree of dispersity of the dye in solution is considered to be an important factor in determining the affinity. M. CLARK.

Behaviour of different starches towards dyestuffs and iodine. I. J. HUEBNER and K. VENKATARAMAN (J. Soc. Dyers and Col., 1926, 42, 110—121).—An investigation, using titration and colorimetric methods, of the amount and rate of absorption of direct, acid, and basic dyes from cold aqueous solutions by ungelatinised granules of maize, potato, rice, sago, tapioca, and wheat starches. Basic dyes are absorbed to a greater extent than acid and direct dyes by starch, the amount of absorption varying with different starches and the resulting shades of the dyed starch being (in several instances) unrelated to the amount of dye absorbed. Starch granules may be uniformly dyed throughout by means of Prussian blue, but they have no affinity for picric acid. Starch dyed with most dyes is fairly fast to washing with water. The absorption by starches of a basic or acid dye from solutions of increasing concentration approaches a maximum but the corresponding absorption of a direct dye is irregular The maximum amounts of Magenta and Methylene Blue absorbed by potato starch and of Crystal Scarlet by wheat starch are 0·923%, 0·473%, and 0·195% respectively. The power of various starches to absorb basic dyes decreases in the order, rice, maize, potato, tapioca, wheat, and sago, but maize and potato, and tapioca and wheat starches respectively frequently interchange places. The rate of absorption of Methylene Blue from aqueous solutions

is nearly complete within a few minutes. The absorption of Benzopurpurine 4B is slightly and considerably increased by the addition of sodium phosphate and sodium chloride respectively. The affinity of all starches except sago for basic dyes is decreased after gelatinisation and drying. Starched and ordinary bleached cotton fabric, absorb Methylene Blue equally, but after dyeing the latter has the deeper shade. Absorption of Methylene Blue by starch is considerably decreased by the presence of alcohol. Starch absorbs copper but not iron salts from aqueous solutions; potato starch absorbs 0·37% of copper sulphate from a 0·2% solution. Starches absorb 0·85—2·66% of tannic acid.

A. J. Hall.

[Printing] white and red discharges on indigo grounds. J. Pokorny (Sealed Notes 2318 and 2328, 24.4 and 22.6.14. Bull. Soc. Ind. Mulhouse, 1925, **91**, 759—761. Report by P. Seyder, *ibid.*, 762—764).—During investigations on a printing paste containing lead chromate, manganese dioxide, and sodium chlorate for obtaining white or coloured discharges on indigo-dyed fabric, Pokorny found that the manganese dioxide and sodium chlorate were not essential for coloured discharges and that white discharges could be obtained without sodium chlorate, but with a smaller proportion of manganese dioxide. For example, in obtaining red discharges, cotton dyed with indigo is mercerised, prepared with β-naphthol, printed with a paste containing 4800 g. of lead chromate, 9325 g. of gumtragacanth + starch thickening, 6575 g. of diazotised *p*-nitroaniline, and 3000 g. of water, then passed during 3—5 sec. through hydrochloric acid of 17° B. (*d* 1·13) at 37°, steamed for 35—40 sec. at 35°, and washed. White discharges are obtained by means of a similar discharge paste containing 4800 g. of lead chromate, 7000 g. of gum tragacanth + starch thickening, 3000 g. of water, and 600 g. of manganese dioxide, and are cleared by soaping. Seyder confirms the excellent results obtained by the processes but indicates their similarity to that mentioned by the B.A.S.F. ("Indigo pur," 179). The discharged portions of the fabric absorb Methylene Blue strongly and appear to contain oxycellulose. A. J. Hall.

Exposure tests with the Osram Pointolite lamp. E. Hochheim and E. Knebel (Melliands Textilber., 1925, **6**, 914—916; Chem. Zentr., 1926, I, 1720).—An arc lamp filled with an inert gas and containing two hemispherical tungsten electrodes which are separated with formation of an arc (when the electric current is first passed through the lamp) by the expansive force produced by the heating of a bimetallic strip, gives light, particularly when the red rays are reduced by passage through a screen of water, which is especially suitable for carrying out fading tests and the comparison of colour tones. The light obtained from the lamp is uniform over considerable periods and is suitable for carrying out quantitative measurements. A. J. Hall.

Patents.

Treating [mercerising] textiles. F. B. Voegeli (U.S.P. 1,577,393, 16.3.26. Appl., 9.2.25). —Textile goods are squeezed and mangled during treatment with hot caustic liquors, then cooled and stretched. A. J. Hall.

Imparting a linen-like character to cotton. H. Matt (E.P. 244,485, 11.12.25. Conv., 11.12.24). —Cotton yarns of less than 60's count and fabrics made therefrom are given a permanent linen-like appearance by treatment for not less than 4 min. at 0—5° with sulphuric acid of 49—50° B. (*d* 1·516—1·532), being subsequently well washed and mercerised under tension by means of caustic soda of 15° B (*d* 1·116) at ordinary temperatures exceeding 0°. A. J. Hall.

Production of colours or lustre on natural or artificial fibrous material. N. V. Nederlandsche Kunstzijdefabriek (F.P. 597,231, 25.4.25. Conv. 30.12.24).—A precipitate of sulphur is produced locally on parts of the material, and dyeing is then carried out. The material may be dipped in a solution of alkali polysulphides and treated with acid, or soaked in a solution of sulphur in an organic solvent with subsequent evaporation of the latter. The presence or absence of sulphur on the fibre results in different tints on dyeing or treatment with metal salts. For viscose silk the desulphurising process is omitted. If viscose is treated with desulphurising agents after dyeing a non-lustrous fibre is obtained. For the production of patterns the sulphur is removed only in parts. B. Fullman.

Bleaching organic materials. R. Hamburger and S. Kaesz (U.S.P. 1,580,136, 13.4.26. Appl., 16.11.23).—See E.P. 209,073; B., 1924, 508.

Means for facilitating the mercerisation or like treatment of yarn samples. P. Caldwell, and Brit. Cotton and Wool Dyers' Assoc., Ltd. (E.P. 250,392, 6.4.25).

Azo dye (U.S.P. 1,576,322).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Absorption of chemical fogs. Determination of sulphur trioxide in moist burner gases. H. Gille (Z. angew. Chem., 1926, **39**, 401—402; cf. Remy, B., 1926, 255).—The sulphur trioxide, together with the sulphur dioxide, produced by roasting pyrites, can be absorbed by passing the dry gases through caustic potash solution, and so estimated. After the gases have passed the washer, the sulphur trioxide appears in the form of a moist fog, which is not completely absorbed by passage through several wash-bottles. It is removed, however, by passing the gases through a 10 cm. length of (glass) wool, from which it can subsequently be recovered by washing, and estimated. The latter procedure, even with moistened wool, fails to remove all the sulphur trioxide from the dry gases. A. B. Manning.

Treatment of crude potassium salts. M. Pappée (Kali, 1925, **19**, 174—179, 217—223, 235—241, 260—265, 275—281, 300—305, 321—324, 356—364,

402—406; Chem. Zentr., 1926, I, 1872).—A review of processes for the extraction of potassium salts from potash deposits is given, including the treatment of the silicious residues. A detailed description is given of large-scale tests on the elutriation process in the extraction of carnallite, using agitating tables, magnesium sulphate, calcium sulphate, and insoluble salts being separated at various stages in the process. The concentration and nature of the washing solutions are important, and the size of the crystals must be considered. Results obtained from various plants are tabulated. Potassium chloride and kieserite can be separated on the agitating table by the use of suitable extracting and washing liquors.

B. W. CLARKE.

Colour reaction of saponin with nitrates. C. A. MITCHELL (Analyst, 1926, **51**, 181).—In the test for nitrates pure commercial saponin (B.D.H.) was successfully used to replace brucine or strychnine. If 1 drop of concentrated sulphuric acid is added to minute quantities of saponin and a nitrate, the blood-red coloration produced appears to be proportional to the amount of nitrate, and apparently to that of the saponin present. D. G. HEWER.

Chemistry of bleaching powder. S. OCHI (Trans. Amer. Electrochem. Soc., 1926, **49**, 137—146. Advance copy; cf. B., 1924, 54, 130, 131, 253).—The development of crystals of $Ca(OCl)Cl,H_2O$ (cf. B., 1923, 603 A) depends on the conditions of manufacture and the nature of the materials; it is favoured by slow chlorination at low temperature in absence of water. In the manufacture it is considered that the hypochlorous and hydrochloric acids, formed by solution of chlorine in the water adhering to the slaked lime, neutralise the lime with the formation of a solid solution of oxychloride, chloride, hypochlorite, and basic oxychloride of calcium from which crystals of $Ca(OCl)Cl,H_2O$ are deposited. All oxygen-forming decompositions of bleaching powder are exothermic; all chlorine-forming decompositions are endothermic. The equilibrium pressures of oxygen and chlorine over $Ca(OCl)Cl,H_2O$ were determined by Nernst's heat theorem. A powder with 42—45% of available chlorine was prepared by chlorination of very finely divided slaked lime. S. K. TWEEDY.

Increasing the internal volume of silica gels by moist heat treatment. H. N. HOLMES, R. W. SULLIVAN, and N. W. METCALF (Ind. Eng. Chem., 1926, **18**, 386—388).—Silica gel prepared by adding ferric chloride to a solution of sodium silicate, and boiling the gelatinous precipitate with hydrochloric acid, has a high absorptive capacity, absorbing 62% of benzene vapour against 32% for a standard silica gel. This is owing to the porous nature of the gel produced by the extraction of ferric hydroxide during the boiling with acid. The absorption can be enormously increased by slow drying of the gel; further, if the partially-dried gel with a water content of about 60%, is submitted to syneresis in a closed vessel for 2—3 weeks, before boiling with acid, a gel which absorbs up to 133% of benzene vapour can be obtained. B. W. CLARKE.

Economics of chlorine. D. A. PRITCHARD (Trans. Amer. Electrochem. Soc., 1926, **49**, 73—85. Advance copy).

PATENTS.

Stabilising liquid hydrocyanic acid. G. H. BUCHANAN, Assr. to AMER. CYANAMID CO. (U.S.P. 1,577,057, 16.3.26. Appl., 16.9.25).—Stabilisation of liquid hydrocyanic acid is effected and corrosion of the iron container prevented by adding 0·1—0·2% of a halogen derivative of the acid, *e.g.*, cyanogen chloride, or its polymer, cyanuric chloride.

R. B. CLARKE.

Distillation of persulphuric acid and of solutions of persulphuric-acid salts. G. BAUM (U.S.P. 1,577,201, 16.3.26. Appl., 5.5.24).—Heat is supplied directly to the liquid by an alternating current of a frequency below 500. H. HOLMES.

Production of barium hydroxide from barium sulphide. A. JAHL (E.P. 249,402, 29.8.25. Conv., 18.4.25).—1000 kg. of porous, crude barium sulphide, containing 65% BaS, is slowly stirred with 250 litres of a solution of barium polysulphide, containing 36% BaS_4. Mixing is effected in such a way that the mass remains dry. After 20—30 min. the mass is leached with boiling water. A concentrated solution of 160 kg. of sodium hydroxide is added to the liquor leaving the leaching vats at 60—65° and on cooling nearly all the barium is deposited as barium hydroxide. If the liquor is cooled without addition of alkali only half the amount of hydroxide is precipitated, but, on passing carbon dioxide into the mother liquor a mixture of barium carbonate and sulphur is obtained. This yields barium sulphide when calcined with carbon. R. B. CLARKE.

Distillation of crude ammoniacal liquor in a column apparatus. C. STILL (G.P. 423,849, 26.9.22).—The distillation is carried out without the use of foreign vapours for heating. The liquor freed in the first column from the greater part of the volatile ammonium compounds, is led into a boiler before treatment with lime. The steam containing carbon dioxide from the boiler is passed into the lower part of the second column, and the hot liquor together with lime is introduced into the upper part. The method offers heat economies. B. FULLMAN.

Evaporation and concentration of [salt] solutions. C. COLLARD (E.P. 227,843, 14.1.25. Conv., 14.1.24).—The process described in E.P. 166,896, may be applied to the concentration and evaporation of salt solutions by using an elongated cylindrical drum divided into compartments by steam-tight partitions. Each compartment has at the bottom an inlet nozzle for the hot solution and steam coming from the heater and sprayer and an outlet pipe to convey the unevaporated solution to the next stage, and at the top a vapour outlet pipe opening into a common main. S. BINNING.

Recovering sodium chloride from saline liquors. A. W. GAUGER and H. H. STORCH, Assrs. to BURNHAM CHEMICAL CO. (U.S.P. 1,576,739, 16.3.26. Appl., 16.2.25).—Brine is evaporated until

sodium chloride, with its impurities, crystallises. The crystals are separated, ground, and purified by washing with the original dilute brine.
C. Irwin.

Preparation of sodium sulphide or similar inorganic fusible chemicals in the form of small lumps. Chem. Fabr. Kunheim & Co. (E.P. 233,316, 17.3.25. Conv., 1.5.24).—Sodium sulphide or similar compounds are obtained in tablet form by allowing drops of the fused salt of the desired size to fall vertically on to a cold hard surface of metal or ebonite, covered if necessary with vaseline. The shape of the tablets is determined by the temperature of the fused salt and the height through which the drops fall.
C. Irwin.

Manufacture of alkali hydrosulphites [hyposulphites]. Farbenfabr. vorm. F. Bayer & Co. (E.P. 247,523, 12.12.25. Conv., 13.2.25).—Considerably increased yields of hyposulphite are obtained from alkali amalgam and bisulphite solution if a portion of the mother liquors be removed periodically or continuously, and replaced by water or a salt solution. R. B. Clarke.

Manufacture of barium nitrate. D. Tyrer (E.P. 248,593, 6.5.25).—Witherite is dissolved in hydrochloric acid and the saturated solution of barium chloride obtained treated with nitric acid (*d* 1·40). Crystalline barium nitrate is precipitated and the mother liquor is returned for use in a further extraction. Much larger yields are obtained in proportion to the materials handled than in the direct treatment of witherite with nitric acid, on account of the low solubility of barium nitrate.
C. Irwin.

Making bleaching earth from slag. Erdwerke München O. Lietzenmayer (E.P. 248,639, 17.8.25. Conv., 25.3.25).—Undried clay is ground to extreme fineness and mixed with sufficient acid to convert impurities into soluble salts. It is then dried to a paste which still contains free acid, lixiviated with water to remove soluble salts, and the sludge dried and ground. C. Irwin.

Manufacture and manipulation of colloidal or semi-colloidal substances, precipitates or sediments and the recovery of the liquid or solid components. Spencer Chapman & Messel, Ltd., and J. B. Liebert (E.P. 249,647, 19.2.25; cf. E.P. 236,087; B., 1925, 671).—A turbid solution of a salt or oxide of titanium, thorium, iron, etc., can be quickly clarified, without settling, by adding a small amount of glue of animal origin, stirring in a light, inert substance which presents a large surface to the solution, *e.g.*, asbestos, and finally filtering. R. B. Clarke.

Article of manufacture. [Calcium silicate derivative.] H. A. Endres, Assr. to Celite Co. (U.S.P. 1,574,380, 23.2.26. Appl., 23.6.25).—60 pts. of finely divided diatomaceous earth are heated at 100° with 56 pts. of lime and 1000 pts. of water for 3 hrs. A vigorous reaction takes place with formation of hydrated calcium silicate, which is calcined at 550—900° preferably in presence of carbon dioxide to remove free lime. The product is a very finely divided powder, the average particle size being 1 micron. It is of value as a filler, a filter aid, and a decolorising agent, and as an ingredient of cements.
T. S. Wheeler.

Purifying aluminium nitrate. T. Mejdell and O. Ravner, Assrs. to Norsk Hydro-Elektrisk Kvaelstofaktieselskab (U.S.P. 1,575,634, 9.3.26. Appl., 27.2.22).—Hot and cold solutions of aluminium nitrate are mixed, the conditions of temperature and concentration being so regulated that crystallisation occurs. Iron compounds remain in solution.
C. Irwin.

Method of making aluminium chloride. A. McD. McAfee, Assr. to Gulf Refining Co. (U.S.P. 1,578,052, 23.3.26. Appl., 1.3.23).—Chlorine is passed over a mixture of carbon and aluminium oxide. The period of induction in the reaction is shortened by mixing an oxide of carbon with the gas until the reaction starts. W. N. Hoyte.

Manufacturing arsenates. D. Lopez (U.S.P. 1,578,150, 23.3.26. Appl., 2.5.24).—A slurry consisting of calcium hydroxide (5% of which is freshly precipitated) and water at 30—50° is continuously circulated, by means of a centrifugal pump, from the bottom of an open tank, with a conical bottom, to a tubular oxidising chamber extending from above the top to the bottom of the tank. Arsenious oxide is added to the slurry in the tank, which is provided with a stirrer, and chlorine gas is passed into the oxidising chamber. Tri-calcium arsenate and scarcely any water-soluble arsenate is formed. Milk of lime is added after completion of the oxidation and a light, fluffy product suitable for use as an insecticide is obtained. R. B. Clarke.

Making oxidised leaden powder [litharge]. C. A. Hall (E.P. 248,602, 26.5.25).—The known process for the preparation of litharge by blowing air through a rotating drum containing lead balls is modified by cooling the drum with water. This enables the speed of the drum and the quantity of lead under treatment to be increased. The temperature within the drum is not allowed to rise above 182°. In a drum 6 ft. diam. and 10 ft. long, making 14 revs. per min., 480 lb. of lead per hr. can be oxidised.
C. Irwin.

Manufacture of iron carbonyl. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 248,683, 14.12.25. Addn. to 244,895; B., 1926, 191).—In the preparation of iron carbonyl by the action of carbon monoxide on iron, pressures below 50 atm., even down to 1 atm., may be used if there is sufficient speed in the gas current to avoid deposition of liquid iron carbonyl on the mass. Reaction gases containing 4—6% of iron carbonyl are obtained. Small amounts of ammonia, methyl alcohol, or formaldehyde accelerate the reaction, but even traces of oxygen or carbon dioxide may render the surface of the iron inactive.
C. Irwin.

[Making] zirconium carbide. M. L. Hartmann, Assr. to Carborundum Co. (U.S.P. 1,576,275, 9.3.26. Appl., 13.3.24).—A zirconium silicate ore is fused in an electric furnace under reducing conditions. C. Irwin.

Extraction of hydrogen contained in industrial gases and chiefly in coke-oven gas. Ammonia (E.P. 242,583, 23.12.24. Conv., 6.11.24).—Hydrogen is separated from coke-oven or other industrial gas, after the preliminary removal of water vapour, carbon dioxide, and heavy hydrocarbons, by condensing the remaining components from the compressed gas, utilising, once the apparatus is in operation, the cold produced by the expansion of the residual hydrogen, and using liquid nitrogen as an additional external source of cold. The gas mixture is cooled successively in two separators, in one of which the cooling agent is liquid methane, and in the other mainly liquid nitrogen and finally expanded hydrogen. The hydrogen so separated is passed through liquid nitrogen, the expansion of the hydrogen being so adjusted that the two gases issue from the apparatus in the correct proportion for the synthesis of ammonia. A. B. Manning.

Making graphite. V. M. Weaver (U.S.P. 1,576,883, 16.3.26. Appl., 2.3.23).—At the bottom of a trough-shaped furnace having an insulating lining of powdered lampblack is placed a slab of carbon, surrounded by a vertically movable frame. The space inside the frame is charged with a mixture of lime and petroleum coke, a layer of the latter covering the main charge. A number of electrodes are arranged above the slab. Fluid calcium carbide is formed at first at the bottom of the furnace but is later converted into graphite and calcium ; the latter volatilises and rises forming fresh carbide. The electrodes and frame are raised to keep pace with the reaction which takes place in zones progressively upward. The graphite finally obtained may be compressed while hot by transferring the whole furnace to a press. R. B. Clarke.

Lime burning process. I. Warner (E.P. 250,045, 15.5.25).—See U.S.P. 1,542,195 ; B., 1925, 631.

Drying calcium hypochlorite compounds. Chem. Fabr. Griesheim-Elektron, Assees. of G. Pistor (U.S.P. 1,572,952, 16.2.26. Appl., 26.3.25).—See E.P. 232,560 ; B., 1925, 714.

Treating crude oxide and carbonate of zinc. W. T. Gidden and W. G. Ragg, Assrs. to Chance and Hunt, Ltd. (U.S.P. 1,579,302, 6.4.26, Appl., 28.11.23).—See E.P. 228,244 ; B., 1925, 241.

Production of ammonia from gases containing hydrogen cyanide. B. F. Halvorsen, Assr. to Norsk Hydroelektrisk Kvaelstofaktieselskab (U.S.P. 1,580,038, 6.4.26, Appl., 16.2.25).—See E.P. 231,134 ; B., 1925, 957.

Manufacture of hydrogen sulphide. F. W. Sperr, jun., Assr. to Koppers Co. (U.S.P. 1,580,451, 13.4.26. Appl., 4.4.21. Renewed 3.7.25).—See E.P. 209,171 ; B., 1924, 173.

Recovery of hydrogen sulphide. F. W. Sperr, jun., Assr. to Koppers Co. (U.S.P. 1,580,452, 13.4.26. Appl., 8.12.21. Renewed 3.7.25).—See E.P. 190,118 ; B., 1924, 173.

Separating hafnium and zirconium. D. Coster and G. von Hevesy, Assrs. to Naaml. Vennoots. Philips' Gloeilampen-Fabr. (U.S.P. 1,580,650, 13.4.26. Appl., 13.3.24).—See E.P. 220,359 ; B., 1924, 869.

Determination of carbon monoxide (U.S.P. 1,578,666).—See II.

Preparation of mixtures of chromates and dichromates (G.P. 420,444).—See IV.

VIII.—GLASS; CERAMICS.

Baroni reaction for neutral glass for pharmaceutical purposes. L. Tirelli (Boll. chim. farm., 1926, **65**, 65—72).—For glass to be used for pharmaceutical purposes and particularly for holding material to be injected, Baroni (Boll. chim. farm., Nov. 30, 1925) has suggested the following test. A vessel of the glass, filled with distilled water and sealed in the flame, is immersed in steam at 2 atm. pressure for 1 hr., and, when cold, re-opened. When treated with 3—4% of its volume of a fresh alcoholic 1% solution of crystallised hæmatoxylin, the water should then exhibit no pink or violet-blue coloration within 24 hrs. The author finds that great care is necessary to ensure that the reagents are pure and to avoid contamination with dust, vapours, etc., from the air. With glass having a basis of silicates, the test gives a sensitive indication of removal of bases or metals, especially copper, from the glass. When, however, the glass contains an excess of borates, negative results leave the issue in doubt. The test should be used in conjunction with, and not instead of, that in which phenolphthalein is employed. It gives no indication of the loss by the glass of neutral salts, such as sodium sulphate, or of acidic residues, such as silica, borosilicic acid, etc., which might affect the keeping properties of special injections like colloidal metals, gelatinous sera, alkaloids, alkaline liquids, etc. T. H. Pope.

Testing for refractoriness and after-contraction, and experiences with refractories in vertical [gas] retorts. T. F. E. Rhead and R. E. Jefferson (Trans. Ceram. Soc., 1925—26, **25**, 6—38).—See B., 1925, 501.

Patents.

Heating metal, glass or other material to render it workable. J. W. Van Meter (U.S.P. 1,572,975, 16.2.26. Appl., 5.6.22; cf. U.S.P. 1,573,784 p. 444).—Sawdust is mixed with zinc, iron, or the like and packed into a glass tube which it is required to bend. On passing chlorine through the tube the heat evolved by the action of the gas on the metal softens the glass. The sawdust is carbonised and forms a packing which prevents distortion of the tube. The latter is cooled and cleaned by

passing compressed air through it. Articles may be heated externally by surrounding them with a mixture of iron and sawdust and treating the latter with chlorine. T. S. Wheeler.

Tunnel kilns. W. L. Hanley, jun. (E.P. 249,265, 26.1.25).—The firing chamber of a tunnel kiln consists of two firing zones separated by a longitudinal partition and heated by furnaces on either side of the chamber. Goods to be fired travel through the kiln on cars on parallel tracks in opposite directions, deflecting walls adjacent to the outer walls protecting the cars from direct heat. The products of combustion are removed through eduction flues, in which dampers are placed so that the temperatures of individual furnaces may be regulated. The firing zones terminate at either end in tunnel sections carrying two parallel tracks, forming combined preheating and cooling zones, in which unfired goods introduced into the kiln are heated by heat from the fired goods from the firing chamber travelling in the opposite direction. This method of kiln construction results in a considerable saving in first cost and operating charges and is specially applicable to the production of large quantities of cheap ware. B. W. Clarke.

Unburned refractory brick. R. H. Youngman (E.P. 250,480, 24.10.25).—See U.S.P. 1,564,394 ; B., 1926, 192.

Fritted quartz filters (E.P. 226,182).—See I.

Furnaces (E.P. 234,049).—See I.

IX.—BUILDING MATERIALS.

Corrosion of concrete. J. R. Baylis (Proc. Amer. Soc. Civ. Eng., 1926, 52, 549—579).—The corrosion of concrete under ordinary conditions appears to depend upon the solubility of calcium carbonate, which is determined by the alkalinity, hydrogen-ion concentration, and salt content of the surrounding water. The alkalinity of a concrete structure, *i.e.*, parts of calcium carbonate dissolved per million, is a good guide as to the state of the concrete ; a value below 200 is an indication that disintegration is liable to occur rapidly. The porosity of the mortar binding the larger aggregate in the concrete is the most important factor in determining the actual rate of disintegration ; a high porosity enables the disintegrating solution to penetrate more rapidly, and also accentuates the destructive influence of freezing. B. W. Clarke.

Durability of cement drain pipe and concrete in alkali soils : Fourth Progess Report (1923). G. M. Williams and I. Furlong (Tech. Papers U.S. Bur. Standards, 1926, 20, [307], 191—240 ; cf. Wig and others, B., 1917, 649).—Concrete drain pipes and concrete blocks even of the best materials and manufacture, are severely attacked by prolonged exposure in soils containing considerable quantities of salts of the sulphate type, but less seriously where salts of the chloride or carbonate type predominate. The durability of cement drain pipe varies with the richness and consistency of the mix, machine-made pipes giving the best results. Wet-mix pipes in general are more resistant to attack than dry-mix pipes, probably owing to decreased permeability. Owing to variations in salt concentration and moisture content from place to place and from season to season in the soil, it is impossible to estimate the maximum concentration to which the test-pieces may have been exposed. Adequate drainage reduces the average concentration of salts in the soil and lessens the rate of attack, and is essential for all concrete work in alkali soils. The presence of reinforcement appears to accelerate disintegration where the permeability of the concrete is high owing to the corrosion and expansion of the metal. The action of the sulphate waters appears to be on the lime set free in the setting of the concrete, producing a compound which occupies a greater volume than that of the original. B. W. Clarke.

Patents.

Cement objects having polished and translucent surfaces. Soc. "Lap," Assees. of S. Seailles (E.P. 234,846, 29.5.25. Conv., 30.5.24).—Special aluminous cements, containing less than 1·5% of iron, *e.g.*, composed essentially of monocalcium aluminate, are used in contact with polished moulds to form articles having a polished and somewhat translucent surface, which may be coloured or decorated. B. W. Clarke.

Production of lasting coloured stains in and upon the surface of cement or other building materials. J. Koebig (U.S.P. 1,577,729, 23.3.26. Appl., 1.11.24).—Two substances capable of interacting with formation of a coloured substance insoluble in water are dissolved in a solvent which will prevent the formation of the insoluble substance. The cement, the surface of which has been rendered porous by treatment with acid and subsequent washing, is impregnated with this solution. On penetrating the cement the solvent evaporates and the coloured insoluble substance is deposited. For instance, the greenish-yellow basic precipitate obtained by mixing aqueous solutions of cupric sulphate and sodium chromate is dissolved in ammonia solution to form the impregnating solution. R. B. Clarke.

Impregnating wood. J. Pintsch A.-G. (E.P. 244,449, 28.11.25. Conv., 12.12.24).—Crude tar from gas producers is a suitable impregnating material for timber, having a low viscosity and high penetrating power at 80°, and a high viscosity at low temperatures, with a relatively high proportion of high-boiling constituents. B. W. Clarke.

Producing homogeneous, mouldable powders from fibrous [asbestos] cements. I. E. and O. E. Lanhoffer, Assrs. to S. J. Dannenberg (U.S.P. 1,580,787, 13.4.26. Appl., 18.3.25).—See E.P. 199,350 ; B., 1924, 383.

Production of artificial stone, stoneware, and the like from boiler slag, dust slag, or ashes.

F. KÖRBER and W. HESSEL (U.S.P. 1,580,906, 13.4.26. Appl., 7.6.24).—See E.P. 218,275; B., 1925, 319.

Utilisation of liquid slag from gas producers etc. (G.P. 423,793).—See II.

Basic carbonate of magnesium (U.S.P. 1,573,603–4).—See VII.

Calcium silicate derivative (U.S.P. 1,574,380).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Qualitative and economic importance of acid electric steel. A. MÜLLER-HAUFF (Stahl u. Eisen, 1926, **46**, 213—218, 289—294).—The acid-lined electric furnace resembles a large crucible and the high quality of the steel produced is due to the action of nascent silicon and the deoxidation and degasification which occur. As carbon is only slowly reduced the scrap used should be as soft as possible. During melting 50—80% of the manganese present is burned away. The materials balance of an acid electric heat is given. The slag plays a less important rôle than in the basic electric furnace. The melting slag amounts to about 4% of the charge. In the deoxidising slag the silica increased up to 75%. The reduction of silica is an important reaction in the acid electric furnace; it is affected by the fluidity and temperature of the slag. A diagram shows the amount of silicon taken up in relation to the extent of the reacting surfaces. The acid electric furnace lining has a good resistance to oxides of manganese and iron, and the life of the roof is 3 to 4 times that of the basic furnace. The silica in the hearth decreases with service. Phosphorus is not reduced and sulphur only slightly during the oxidation period. Mechanical tests on steels with increasing phosphorus content show that not until 0·066% P is reached does deterioration occur in the reduction of area and impact test, the favourable structure compensating for high phosphorus content. A number of acid and basic carbon tool steels were hardened at 750—800°. The acid steel though not superior in mechanical strength showed greater regularity and was less sensitive to hardening temperature. Static and dynamic tensile tests and impact and repeated impact tests were made on medium carbon and nickel-chromium steels made in the acid electric and basic Siemens–Martin and electric furnaces. The electric steels were superior to the open-hearth steels and acid steel was better than basic as regards yield point, maximum strength, and repeated impact test, but slightly inferior in reduction of area. The acid electric process is expensive since pure scrap must be used for ingots, and is principally used for castings where purity need not be so high. The time required for melting and alloying high-carbon and nickel-chromium steels is half that for the basic furnace, and there is less loss of alloy elements in high-chromium and tungsten steels. The combination of a refining furnace with an acid electric furnace is recommended as a quick method of working for high final quality. The cost for lining and upkeep of the acid electric furnace is less than for the basic. As the silicon content of acid steel tends to increase after deoxidation little time is available for the addition of ferro-alloys. T. H. BURNHAM.

Present position of the basic hearth refining process in comparison with the Thomas process. F. BERNHARDT (Stahl u. Eisen, 1926, **46**, 1—7, 39—44, 73—78, 137—142).—The pig iron-ore process is divided into two periods—refining and finishing. It may be carried out continuously or by heats, in a stationary or tipping furnace, and in one or more furnaces. The relative merits of these features are considered with reference to the Hoesch, Dortmund Union, Witkowitz–Talbot, and Königshütte processes. In the last-named refining is effected continuously in a tipping furnace and the finishing process is performed in a separate furnace. The metallurgical details of the process are given in a table including slag analyses at the various stages of operation. Dephosphorisation and decarbonisation take place in the finishing furnace without danger of rephosphorisation. For the same output the Witkowitz and Königshütte processes seem equally economical, but the latter possesses the advantage of better metallurgical control. Under all circumstances the use of tipping furnaces is advantageous. A comparison is made of Siemens–Martin with electric furnace and crucible steel. For certain purposes where the highest degree of deoxidation and degasification is required, crucible steel is unequalled, but steel can be made in the Martin furnace of a high degree of purity, and this furnace is now metallurgically equal to and more economical than the electric furnace. From the heat economy point of view, when the blast furnace and steel works are considered as a unit, the converter process does not excel the Martin furnace and as regards materials it is economically inferior. The ore and coke consumption and slag production are compared for the Thomas and the pig iron-ore and pig iron-scrap processes. Based on pre-war figures a simple formula has been deduced which shows when the Thomas and when the Siemens–Martin process is more advantageous, depending on local conditions. The cost of the construction of a Siemens–Martin installation is higher than that for a Thomas works of the same output, but in the first case the blast-furnace installation is smaller so that the total cost is approximately the same. T. H. BURNHAM.

Heat treatment and testing of chromium magnet steel. E. H. SCHULZ and W. JENGE (Stahl u. Eisen, 1926, **46**, 11—13).—The magnetic properties of chromium steel depend on the hardening temperature, the time the metal is held thereat, the rate of heating, and the size of the specimen. Tests on a steel containing 1% C and 2% Cr showed that the product of remanence and coercive force was a maximum for 10 min. heating at a hardening temperature of 800°. The theories of Gumlich (cf. B., 1922, 143 A) and Evershed (J. Inst. Elect. Eng., 1920, **58**, 780) for the deterioration of magnetic properties

with continued heating at the hardening temperature are discussed. The authors consider that owing to the slow diffusion of the carbon in these steels the solid solution is not homogeneous, and on quenching the change starts at numerous centres in the interior of the crystals. The longer the temperature is maintained the less are the differences in concentration and the fewer the number of nuclei at which the change can start. The optimum time of heating should be determined in each case and for greater certainty should be checked by electrical resistance determinations. The finer the grain and distribution of the carbide before hardening the better are the magnetic properties, so that unnecessary annealing should be avoided. Simultaneously with the carbide going into solution, the deleterious processes of diffusion and grain growth set in. Undissolved carbide inclusions are not necessarily deleterious unless above a certain size. Although a high hardening temperature increases the rapidity of solution it must not be above a certain limit, as there is a greater risk of hardening cracks and of the formation of austenite. The finest distribution of carbides also gives the optimum magnetic properties to cobalt steel. For a steel containing 1·09%C, 16·0% Co, 8·8% Cr, and 2·4% Mo, the following treatment gave satisfactory results :—heated to 1200° and cooled in air, tempered at 750°, and subsequently hardened at 1000° and cooled in air. No definite conclusions on magnetic properties can be drawn from chemical composition. Maximum values can only be obtained in the absence of flaws in the steel and after the optimum heat treatment. Characteristic curves should be determined statistically, not from a single series of tests.

T. H. Burnham.

Pickling iron with hydrochloric and sulphuric acids. H. Bablik (Stahl u. Eisen, 1926, **46**, 218—222).—To determine which acid is the more efficient, discs of mild steel were immersed in 5% hydrochloric and sulphuric acids. Hydrogen produced by attack of the subjacent iron caused mechanical flaking of the scale, this action being more effective in the case of sulphuric than of hydrochloric acid. The specific pickling effect is greater for sulphuric acid and less acid is required, but the pickling period is longer. The pickling velocity of hydrochloric acid rises continuously with increasing concentration; for sulphuric acid the maximum is at 25% strength. At 60° the pickling velocity of both acids increased tenfold. For practical purposes a warm bath of sulphuric acid is required. The pickling velocity was decreased by the presence of reaction products. The amount of chlorides left on the surfaces, which have to be removed by washing is less than in the case of sulphates. Boiling water should be used for complete removal. Pickling with hydrochloric acid is handier and simpler but more expensive. The solution velocity of iron wire and sheet in acids of different concentrations was determined with and without the addition of 0·1 to 0·3% of Vogel's "VBZ" reagent. As only those reactions which liberate hydrogen were retarded by the VBZ reagent, it is thought that its action is not due to adsorption but to its effects on the surface tension and viscosity of the pickling reagents which produce a mechanical hindrance to hydrogen evolution. By the use of Vogel's reagent a saving in iron and acid is effected and a smoother surface imparted to the iron.

T. H. Burnham.

Iron–nickel equilibrium diagram. L. Grenet (Rev. Mét., 1926, **23**, 143—154; cf. B., 1925, 808).—The nature of transformations is discussed and the α–γ transformations in the iron–carbon, iron–nickel, and iron–cobalt series are considered in the light of the existence of a two-phase region between the α and γ phases.

M. Cook.

Cementation of ferrous alloys by chromium. J. Laissus (Rev. Mét., 1926, **23**, 155—178; cf. B., 1925, 635).—On heating polished steel specimens containing 0·15% C in powdered ferrochrome the outside bright layer of carbide increases in thickness as the cementation temperature is raised, the intermediate zone of eutectic increases, and the interior zone of solid solution diminishes until after heating, for 5 hrs. at 1200° it entirely disappears. As the duration of heating is increased the solid solution formed reachs a maximum at 1100° after 5 hrs. and the carbide layer increases linearly. The process of cementation of electrolytic iron is similar to that of 0·15% C steel but the absence of carbon favours the formation of the carbide layer. Raising the carbon content to 0·4% and 0·85% causes a diminution of the solid solution zone, which is practically nil at 0·4% C, and lowers the temperature of formation of the carbide layer which decreases in thickness. The thickness of the layer formed in grey iron is greater than that formed in 0·4 and 0·85% C steels. With steel containing 13% Cr and 0·15% C the bright outside layer contains particles of carbide disseminated through it. Increasing the carbon content of the ferrochrome causes a decrease in the outer carbide layer until it reaches an approximately constant value, the eutectic gradually disappears, and the solid solution increases. No zone of solid solution exists after cementing with powdered electrolytic chromium, which produces the greatest thickness of carbide layer at 1200°. The addition of 45% of aluminium to the powdered chromium results in a thinner layer being formed.

M. Cook.

Heat treatment of aluminium–copper alloys. L. Guillet and J. Galibourg (Rev. Mét., 1926, **23**, 179—190; cf. B., 1926, 92).—The change in hardness, by quenching from 525° and tempering at 100°, 200°, and 300°, of a series of alloys containing from 5 to 50% Cu, has been studied. Owing to partial fusion at 525° alloys containing 45 and 50% Cu were quenched from 450°. The hardness immediately after quenching, for the whole series of alloys, was greater than that of alloys heated to 450° and slowly cooled, and in the case of the 20% Cu alloy the value was doubled. After annealing for 1 hr. at 100° immediately after quenching, the hardness was slightly less than that of quenched alloys, and annealing at 200° for 1 hr. caused further softening. The hardness was increased by prolonging the annealing

at 100°, the maximum hardness for this tempering temperature being attained in 48 hrs. The electrical resistivity reached a maximum in the quenched alloys after 48 hrs. at ordinary temperature, and fell as the tempering temperature was raised. In general the maximum hardness was attained at tempering temperatures which were lower as the copper content increased. The dimensions of a piston of 13% Cu alloy showed no change on heating for periods of 2 hrs. at 250° after water-quenching from 475°. The hardness became constant at about 84 (Brinell) after the fourth reheating. The addition of 1% Mg still further increased the hardness of the alloys after heat treatment. M. COOK.

Measuring polarisation and resistance of electrolytes. HARING.—See XI.

PATENTS.

Electrolytically treating ferrous metal objects. E. POTTER. From MADSENELL CORP. (E.P. 248,797, 11.9.24).—Prior to coating iron or steel with another metal, preferably nickel, either by electrodeposition or otherwise, it is made the anode in a cell containing sulphuric acid of at least 84·5% strength and a lead cathode. The initial voltage is 6—8 volts and the flow of current 20—50 amp. per sq. ft. The treatment is continued for about 3 min. until the flow of current drops to $^1/_5$ to $^1/_{10}$ of its initial value. After washing, the anode is coated with nickel by electrodeposition preferably within the critical period of about 1 min. required for maximum adherence. M. COOK.

Steel-like alloy for church bells. F. LANGE and P. STEINEL (G.P. 421,576, 10.2.22).—A steel-like alloy for church bells consists of iron with 2·8% C, 0·31% Mn, 0·64% P, 0·08% S, and 0·24% Si. A. R. POWELL.

"Tempering" metals and other materials. J. W. VAN METER (U.S.P. 1,573,784, 16.2.26. Appl., 9.6.22).—The method described in U.S.P. 1,572,975 (p. 440) is applied to "tempering" steel articles by the action of cyanogen. A cyanide is added to a mixture of sawdust and iron, and the heat generated by the action of chlorine liberates cyanogen from the cyanide. Alternatively steel heated by the method described may be treated with cyanogen gas under pressure. T. S. WHEELER.

Conversion of cuprous materials. M. H. MERRISS, Assr. to NICHOLS COPPER CO. (U.S.P. 1,576,776, 16.3.26. Appl., 1.8.25).—Blister copper is obtained from black copper by heating the latter until at least partially melted and blowing air in regulated quantity and under controlled pressure through the mass to effect rapid oxidation of the impurities. M. COOK.

Extracting metallic values from ore concentrates and the like. H. FLECK and W. G. HALDANE (U.S.P. 1,577,217, 16.3.26. Appl., 8.8.22).—Ore concentrates or slimes are mixed with a reagent which will convert the silica present into a compound that will volatilise readily and this compound is then allowed to evaporate. A. R. POWELL.

Ore concentration. C. P. LEWIS, Assr. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,577,328, 16.3.26. Appl., 26.7.20).—Ore pulp is mixed with a flotation agent containing a heavy hydrocarbon carried in a hydrocarbon of the benzene series, and gaseous bubbles are brought into contact with the solid particles in the pulp so as to produce a mineral-bearing froth which is separated. M. COOK.

Recovering rare metals from their ores. K. B. THEWS. Assr. to W. A. J. BELL (U.S.P. 1,577,411 16.3.26. Appl., 12.7.21).—Carnotite ore is heated in the presence of a reducing agent and afterwards treated with an acid solvent to dissolve the vanadium, uranium, and radium present. M. COOK.

Electrolytic [metal] separation apparatus. A. DASSBACH, Assr. to IRVINGTON SMELTING and REFINING WORKS (U.S.P. 1,577,898, 23.3.26. Appl., 11.7.24).—An apparatus for electrolytic separation of metals comprises a number of dissolving vats in which anodes and cathodes are supported. Scrapers interposed between the cathodes and anodes are supported on detachable bars mounted upon separate movable carriages. Detachable connexions are provided between the carriages, and means to permit the removal of each carriage as a unit. J. S. G. THOMAS.

White-gold alloy. E. M. WISE, Assr. to WADSWORTH WATCH CASE CO. (U.S.P. 1,577,995, 23.3.26. Appl., 28.10.25).—The alloy contains 30—80% Au, and nickel, copper, and zinc in the relative proportions of 33·0—53·1 pts. Ni, 26·4—46·5 pts. Cu, and 10·6—30·7 pts. Zn. A. W. HOTHERSALL.

Aluminium alloy. T. S. FULLER and D. BASCH, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,578,979, 30.3.26. Appl., 18.12.24).—The alloy contains zinc, 3—8%, magnesium, 0·25—1·5%, and the remainder aluminium. L. M. CLARK.

Purifying molten metals. H. G. LAPSLEY, Assr. to METALS REFINING CORP. (U.S.P. 1,578,044, 23.3.26. Appl., 4.8.25).—A chemical compound of aluminium and boron is added to the molten metal. A. W. HOTHERSALL.

Recovery of metals [copper, gold, silver] from their ores. H. V. WELCH, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,578,618, 30.3.26. Appl., 12.11.23).—Ores containing gold, silver, and copper are treated with a cyanide solution, and the metals precipitated by a suitable precipitant. A soluble cyanide is regenerated in the solution, which is then used cyclically, the strength of the solution being such that during precipitation of the copper the solution contains more than 4 mols. of cyanide, calculated as sodium cyanide, per 1 mol. of copper in the solution. J. S. G. THOMAS.

Separating metal from metalliferous material. L. E. WEMPLE (U.S.P. 1,578,694, 30.3.26. Appl.

16.11.23).—A mixture of metalliferous material, solid carbonaceous fuel, and flux, in proportions sufficient wholly to reduce the material, is blown into a heated furnace chamber, the temperature of which is maintained high enough to volatilise the metal produced. The vaporised metal passes through a tortuous passage, and during its travel is oxidised, the oxidised metal finally being collected.

A. B. Manning.

Reduction of metal oxides. E. B. Pratt (Can. P. 250,234, 12.6.24).—Mixtures of hydrocarbons with metal oxides, or material containing them, especially iron ore, are heated in tubes of refractory material, to a temperature at which the hydrocarbons are decomposed with formation of carbon, and reduction is completed by heating the material in an electrically heated crucible. L. A. Coles.

Apparatus for production of electrolytic iron. E. Duhme, Assr. to Siemens & Halske A.-G. (U.S.P. 1,570,212, 19.1.26. Appl., 17.6.24).—See G.P. 418,139; B., 1926, 163.

Apparatus for the manufacture of earth metals. H. Dolter (U.S.P. 1,580,469, 13.4.26. Appl., 27.10.24).—See E.P. 240,163; B., 1925, 996.

Alloy. P. Girin, Assr. to Soc. Anon. de Commentry, Fourchambault & Decazeville (U.S.P. 1,580,662, 13.4.26. Appl., 16.4.19).—See E.P. 127,243; B., 1920, 412 a.

Apparatus for galvanising iron plates by the lead-zinc process. R. Passeker (E.P. 241,226, 12.10.25. Conv., 11.10.24).

Annealing and similar furnaces. H. Grünewald (E.P. 250,373, 6.3.25).

Roasting furnace (U.S.P. 1,576,033).—See I.

Purifying gas-house liquor (U.S.P. 1,573,169).—See II.

Heating metal (U.S.P. 1,572,975).—See VIII.

XI.—ELECTROTECHNICS.

Dependence of electromotive force of [lead] accumulators upon acid strength. K. Schreber (Z. Elektrochem., 1926, 32, 143—149).—Two accumulators opposed to each other and containing unequal strengths of sulphuric acid tend to equalise their acid concentration during discharge. The E.M.F. difference is calculated by considering the work done in a reversible cycle in which the richer acid is diluted by condensation of water vapour, and the weaker acid is concentrated by evaporation. The theoretical values are different, depending on whether it is assumed, with Faraday, that the vapour arising from the acid solution has the same temperature as that of the vapour from the pure solvent at the same pressure, or whether, with Gay Lussac, it is supposed that the vapour has the same temperature as the solution. The work in the latter case is greater. Comparison is made between theory and experiment, using results of Dolezalek and Thibaut. It is found that Faraday's assumption leads to satisfactory agreement within the experimental error, while, with Gay Lussac's assumption, this is not the case. C. H. D. Clark.

Small mercury-vapour lamp for laboratory use. W. Clark (Phot. J., 1926, 66, 185—186).—The lamp consists of a quartz or water-jacketed glass test tube, with about 1 cm. of clean mercury at the bottom, and a carbon rod down the tube with its square-cut end 2 mm. or so above the mercury surface. Contact with the mercury is made by a wire down the inside of the tube, and the carbon rod is held in position by an asbestos string wad. The arc burns with a current of from 0·3 to 3 amp. Carbon should be negative to avoid a continuous background in spectrum work. The lamp is used for calibrating spectrographs and preparing wave-length scales. W. Clark.

Laboratory [electric] furnace for high temperatures. J. and W. B. Mitchell (Trans. Ceram. Soc., 1925–26, 25, 39—50).—See B., 1925, 511.

See also pages 426, **Electric ignition of firedamp** (Wheeler). 427, **Autoxidation of mineral oils** (Marcusson and Bauerschäfer). 452, **Test for rubber insulation** (Hippensteel).

Patents.

Non-sagging spiral filaments of highly refractory metal [for incandescence lamps]. N.V. Philips' Gloeilampenfabr. (E.P. 234,489, 20.5.25. Conv., 24.5.24).—Tungsten wire is wound into a spiral and heated to a temperature exceeding 1500°, which causes recrystallisation and prevents the filament from subsequently sagging in use, the sagging of helical wires in incandescence lamps being due to recrystallisation, which occurs principally during the first few hours of burning. The wire may be wound on a refractory core such as molybdenum or tantalum, which is removed by chemical treatment after the annealing operation. M. Cook.

Gas-filled electric lamps. Gen. Electric Co., Assees. of Patent-Treuhand-Ges. für Elekt. Glühlampen m.b.H. (E.P. 248,680, 28.11.25. Conv., 18.3.25).—The incandescent body or electrodes of an electric lamp may be prevented from volatilising to any appreciable extent by adding small quantities of hydrogen and hydrogen halide to the inert gas used for filling the lamp. The hydrogen and hydrogen halide mixture may be added direct to the filling, or suitable substances such as a mixture of hydrogen and tungsten chloride may be introduced, whereby hydrogen halide is produced when the lamp is running, tungsten being deposited on the filament. Alternatively, hydrogen halide alone may be introduced; this attacks the filament, hydrogen being liberated. This filling raises the average life of the

lamp and makes it more uniform; also, the metal sputtered from the filament is transformed into halides which are volatile at the temperature of the bulb and, if deposited on the bulb, produce only a translucent layer. The danger of short-circuiting is also diminished. M. E. NOTTAGE.

Incandescence [electric] lamp. R. E. MYERS, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,572,670, 9.2.26. Appl., 25.6.21).—If glass free from lead, *e.g.*, lime glass, be used for the manufacture of hydrogen-filled incandescence lamps, no blackening of the glass occurs during use. T. S. WHEELER.

Increasing the ultra-violet radiation from mercury vapour lamps made of quartz. H. GEORGE (E.P. 234,834, 29.5.25. Conv., 30.5.24).—The emission in the ultra-violet region is much increased when the lamp is supplied with alternating current at high frequency and high tension. M. E. NOTTAGE.

Mercury vapour lamps. J. NISBET (E.P. 248,796, 11.9.24).—The patent relates to improvements in the design of mercury vapour lamps, especially for projecting pictures from transparencies or opaque objects on to a screen. M. E. NOTTAGE.

Electric gas purifier plants. SIEMENS-SCHUCKERTWERKE GES. M.B.H. (E.P. 240,844, 30.9.25. Conv., 1.10.24).—Dust from the electrodes of gas purifying plants is collected in pockets provided with inclined floors and leading to a common receptacle, arranged under each collecting electrode, spaces being left between them through which the gas to be purified can pass up to the electrodes. M. E. NOTTAGE.

Devices for the chemical treatment of gases. METROPOLITAN-VICKERS ELECTRICAL CO., LTD., Assees. of L. H. HILL (E.P. 241,547, 9.10.25. Conv., 14.10.24).—A device for the chemical treatment of gases, to be used more especially in removing water or other deleterious substances from the air or gas drawn into oil-insulated transformers during "in-breathing," comprises a movable container for the active chemical, supported within a casing through which the gas passes, the relative position of container and casing in use indicating the condition or degree of exhaustion of the active chemical. J. S. G. THOMAS.

Positive electrodes for electric batteries. SOC. ANON. LE CARBONE (E.P. 243,300, 15.12.24. Conv., 19.11.24; cf. E.P. 226,769; B., 1925, 555).—The efficiency of the depolarising medium of positive electrodes may be maintained by protecting it with an external covering of pectisable colloid, impermeable to liquids but permeable to gases. The depolarising medium, which may consist of a mixture of manganese dioxide, plumbago, and wood charcoal, moistened with a solution of ammonium chloride, is compressed on to the carbon positive electrodes forming channels, which permit of the access of the outer air to the interior of the composition. The liquid contained in the pores is then evaporated either by stoving at 150° or by prolonged exposure to air, after which the external surface of the depolariser is covered with a colloidal pectised coating by immersing in or coating with a colloidal solution. The depolarising composition may also be contained either in a bag or in a porous pot open at the top, the colloidal covering being applied to the inner face of the vessel; or the grains of powder may be individually covered with a colloidal coating. M. E. NOTTAGE.

Electric cells. C. GAISER (E.P. 248,942, 8.4.25).—The patent relates to various modifications of cells of the type in which the electrodes have the form of bands or threads which are gradually brought into action by contact of part with the electrolyte while the remaining part is kept outside, preferably wound up and is unwound according to requirements. A layer of absorbent material which may be impregnated with an electrolyte in dry form is placed between the electrodes. The positive electrodes preferably consist of magnesium or of a metal more electro-positive than magnesium, the negative electrodes of oxides such as copper oxide or lead peroxide, and the electrolyte of acid or alkaline compounds of magnesium. M. E. NOTTAGE.

Electrolytes for lead accumulators. A. J. A. DE LA PORTE (E.P. 249,016, 28.8.25).—An electrolyte for lead accumulators which has a great regenerative capacity and can be used even in deteriorated accumulators consists of a solution of ammonium alum in dilute sulphuric acid in the proportions: 16 kg. of sulphuric acid, *d* 1·835, 30 litres of distilled water, and 2·5 kg. of ammonium alum; or 25 kg. of sulphuric acid, *d* 1·835—1·837, 80 litres of water, and 5 kg. of alum. M. E. NOTTAGE.

Electric-furnace system. J. W. HARSCH, Assr. to LEEDS & NORTHRUP CO. (U.S.P. 1,578,027, 23.3.26. Appl., 20.2.25).—The temperature of the chamber of an electric furnace is raised rapidly, and air simultaneously circulated within the chamber, the direction of circulation being reversed at intervals which are short compared with the time taken by the chamber to attain the desired temperature. J. S. G. THOMAS.

Resistance element. W. OTTO, Assr. to NEDERLANDSCHE TECHNISCHE HANDEL MAATS. "GIRO" (U.S.P. 1,577,981, 23.3.26. Appl., 14.2.24).—An electric resistance comprises two portions composed of a mixture of finely divided graphite and at least partially vulcanised rubber in different proportions, the graphite in at least one of the portions being embedded in the rubber. J. S. G. THOMAS.

Preservation of oil used in electrical apparatus. MASCHINENFABR. OERLIKON (G.P. 425,051, 6.5.24. Conv., 14.4.24).—Acids formed in oil contained in electrical apparatus are neutralised without the oil being attacked, by maintaining the oil in contact with "octahedral" borax, $Na_2B_4O_7,5H_2O$. For example, textile material which has been steeped

in hot, saturated borax solution and dried is suspended in the oil. L. A. Coles.

Electrical treatment of liquids. E. Anderson, Assr. to Internat. Precipitation Co. (U.S.P. 1,578,624, 30.3.26. Appl., 20.11.23).—A colloidal suspension of a material dispersed in a poorly conducting liquid is subjected to the action of a silent electric discharge produced in a gas in contact with the liquid, whereby the suspended material is separated from the liquid. J. S. G. Thomas.

Insulating bodies and method of producing them. C. L. Dawes and W. A. Boughton, Assrs. to New England Mica Co. (U.S.P. 1,578,812–3, 30.3.26. Appl., 26.10.22).—(a) Mica fragments are mixed with an inorganic binding material, which at atmospheric pressure becomes fluid at a temperature substantially as high as the disintegration temperature of mica, and the mixture is heated to a temperature below that at which mica disintegrates, pressure being applied to cause the binding material to flow at that temperature. (b) A mixture of mica fragments and an alkali silicate containing substantially no water is subjected to pressure.

J. S. G. Thomas.

Electric furnaces. W. A. Loke (U.S.P. 1,580,060, 6.4.26. Appl., 2.6.25).—See E.P. 239,249; B., 1925, 886.

Electrode connexions for electric furnaces. M. McGee (E.P. 250,002, 21.3.25).

See also pages 438, **Distillation of persulphuric acid** (U.S.P. 1,577,201). 440, **Graphite** (U.S.P. 1,576,883). 444, **Electrolytically treating ferrous metals** (E.P. 248,797); **Electrolytic apparatus** (U.S.P. 1,577,898). 456, **Purification of glue and gelatin** (U.S.P. 1,577,660).

XII.—FATS; OILS; WAXES.

Bromometric examination of fats. H. P. Kaufmann (Z. Unters. Lebensm., 1926, **51**, 3—14; cf. B., 1925, 302).—The activity of bromine in solution in various solvents is discussed and a 0·1 *N*-solution in absolute methyl alcohol saturated with sodium bromide is recommended for the determination of the iodine value of fats. Such a solution, probably due to the formation of sodium tribromide, is very stable and loses only 1% of its active bromine in 56 days. It is almost odourless and can be measured with a pipette without danger. The determination is simply and quickly performed with an accuracy comparing favourably with the Hanus method. E. H. Sharples.

Use of thiocyanogen in fat analysis. H. P. Kaufmann (Z. Unters. Lebensm., 1926, **51**, 15—27; cf. B., 1926, 165).—The "thiocyanometric" iodine values of mixtures of fats have been determined. From a knowledge of this value and the usual bromometric iodine value of a mixture of three fats and its constituents the composition of the mixture may be calculated. For a mixture of two fats, the "thiocyanometric" iodine value alone offers no advantage over the usual iodine value owing to the smaller difference in the values of the single components. E. H. Sharples.

Rapid method of determining the iodine value with iodine and alcohol. W. Austen (Pharm. Zentr., 1926, **67**, 209—210).—In carrying out the method of Margosches, Hinner, and Friedmann (B., 1924, 639) it is of importance that 200 c.c. of water be added to the fat solution, and that the minimum time of contact be 6 min. Observing these precautions, lard, beef tallow, and linseed oil gave iodine values of 58, 35·5, and 172·8 against the Hübl values of 58, 36·4, and 173·4 respectively.

A. Rayner.

New value for milk-fat. J. Kuhlmann and J. Grossfeld (Z. Unters. Lebensm., 1926, **51**, 31—42).—The "butyric acid value" is a modification of the Reichert-Meissl value and is a measure of the water-soluble volatile fatty acids of lower molecular weight than caprylic acid (coconut oil fatty acids). 5 g. of fat are saponified with 2 c.c. of potassium hydroxide solution (75%) and 10 c.c. of glycerol, and the soap solution is diluted with 100 c.c. of water at 100°. The mixture is cooled to 20° and 50 c.c. of dilute sulphuric acid (2·5%), 15 g. of anhydrous sodium sulphate, 10 c.c. of coconut oil soap solution (100 g. of coconut oil saponified with 100 g. of glycerol and 40 c.c. of 75% potassium hydroxide solution and the solution diluted to 1 litre with water), and about 0·1 g. of kieselguhr, are added. After shaking for 10 min. the liquid is filtered and 125 c.c. of the filtrate are diluted with 50 c.c. of water and distilled with the usual precautions. The first 110 c.c. of the distillate, which contain about 96% of the total butyric acid, are directly titrated with 0·1 *N*-alkali and from the result is subtracted that given by a blank without fat. The difference, multiplied by 1·4, is the butyric acid value. The value for coconut-oil is 0·9 and for butter-fat 20, this latter being directly proportional to the Reichert-Meissl value. By the application of small corrections for coconut oil, the amount of milk-fat in admixture with other fats can be calculated from the butyric acid value and the saponification value to within an accuracy of 1%. E. H. Sharples.

Simplified determination of milk fat in fatty mixtures. J. Grossfeld (Z. Deuts. Oel- u. Fett-Ind., 1926, **46**, 49—51, 65—66, 84—85, 97—99, 114—115, 130—132; cf. Kuhlmann and Grossfeld, B., 1926, 165).—The results obtained by the method of Bertram, Bos, and Verhagen (*loc. cit.*), and the distillation method of Gilmour (B., 1925, 564), are compared and the principle of these methods extended and made more sensitive by the determination of saponification value and butyric acid value. The ratio between the factors for milk fat and coconut oil is 3·8 for the Reichert-Meissl value, 12 for the "B" value, 20 for the Gilmour distillation value, and 81 for the butyric acid value (cf. preceding abstract). The probable content of milk fat, *M*, for a mixture having the butyric acid

value, b, is given by the formula $M=5{\cdot}115b-0{\cdot}085v$, where v equals the saponification value minus 200. A table is given for reading off this value. The method gave butyric acid values from 0·0 to 0·6, with saponification values of 194 to 223, from which may be calculated a milk fat content of 0—1·1%, or an average of 0·5%, when applied to several sorts of commercial margarine. On the contrary, butyric acid values of 18·6 to 23·0 (corresponding to butyric acid contents of 3·28 to 4·05%), or an average of 20·3, were obtained with various samples of commercial butter. This corresponds to an average Reichert-Meissl value of 27·3. The average conversion factor for the calculation of the butyric acid value from the Reichert-Meissl value was found to be 0·745. A. RAYNER.

China wood [tung] oil. A. EIBNER and J. SCHWAIGER (Chem. Umschau, 1926, **33**, 77—85).—Mainly a summary and critical review of work published during the past year or two. Reference is made to the isomerisation of α-elæostearic acid on bromination and a few experimental results are quoted. H. M. LANGTON.

Data used in graph for beeswax. A. A. WEIR (Analyst, 1926, **51**, 181; cf. B., 1925, 813).—The limits taken for pure beeswax in the graph were:—d 0·976—0·941; m.p., 60·5—66·5°; n_D, 43·2—45·7; iodine value, 5·42—17·1; saponif. value, 87—106; acid value, 16·7—23·6; ester value, 65·9—85; ratio value, 2·8—4·5; and unsaponif. matter, 48—53%. D. G. HEWER.

Alcohol, acetic acid, and other by-products from arachis nut [pea-nut] shells. G. DE BELSUNCE (Bull. Mat. Grasses, 1926, 1—3).—The Meunier process (B., 1922, 212 A; 1923, 25 A) for the hydrolysis of cellulosic materials with dilute sulphuric acid and high-pressure steam is described. Two experiments with pea-nut shells, one lasting for 30 min. and the other for 60 min., gave the following respective amounts of products calculated on the dry material:—Sugar, 21·8%, 15·8%; acetic acid 2·26%, 4·38%; furfuraldehyde 2·42%, 2·66%; acetone 0·155%, 0·47%; other substances soluble in ether 0·755%, 1·68%. The sugar is entirely fermentable and the residue, consisting principally of lignin, has sufficient heating value to fulfil all the fuel requirements of the process. E. H. SHARPLES.

Activated fuller's earth. O. ECKART (Z. angew. Chem., 1926, **39**, 332—334).—During the bleaching of fatty oils by means of activated fuller's earth the acidity of the oil is increased due to hydrolysis of the glycerides by traces of acid in the earth; this increase is more marked if the earth is moist than after drying. Neutralisation of the acidity of the bleaching agent by treatment with alkaline solutions results in the deposition of thin films of ferric and aluminium hydroxides on the surface of the particles of earth with a consequent diminution, or even total loss, of bleaching power. Addition of calcium carbonate to fuller's earth, forming the so-called "neutral earth," does not prevent the increase in the acidity of the oil after treatment, owing to the sluggish action of the carbonate in an oily medium. A marked diminution in the increase of acidity is, however, produced by the addition of calcium hydroxide equivalent to ten times the acidity of the earth; this addition must be made when the earth is mixed with the oil and not previously. A short time of contact is essential, otherwise the colouring matter again becomes dispersed in the oil; in most cases treatment for 10 min. suffices. Fuller's earth is improved if heated to 400—450° before use and then allowed to adsorb moisture from the air; without the moisture its bleaching power is reduced 10%. Heating above 450° entirely destroys the ability of fuller's earth to remove the colour from oils. A. R. POWELL.

Colour of oils etc. FONROBERT and PALLAUF.—See XIII.

Drying of fatty oils. AUER. WOLFF.—See XIII.

"Voltol-factice." HOCK.—See XIV.

PATENTS.

Extracting oil, gelatigenous water and solid material from whale blubber, whale flesh, fish products, and similar raw materials. K. HOLTER and S. THUNE (E.P. 232,601, 15.4.25. Conv., 16.4.24).—The raw material is fed under pressure down a shaft on to the surface of a revolving grindstone enclosed in a chamber, the temperature of which may be raised by the admission of water or steam. The moist disintegrated material is removed from the base of the chamber by a helical conveyor and passed into a horizontal screening chamber where the main part of the oil and gelatigenous water is removed. The screening chamber is provided with a tapered portion near its outlet and, aided by the conveyor, the material is compressed, thus expelling the rest of the liquid. The temperature of the screening chamber may be controlled by the introduction of steam through the perforated hollow shaft of the conveyor. E. H. SHARPLES.

Neutralising and bleaching oils under heat and vacuum conditions. E. BATAILLE (E.P. 233,345, 29.4.25. Conv., 30.4.24).—The oil is accurately and automatically measured, treated with a measured quantity of neutralising and/or bleaching agent, and drawn by suction into a "beater," *i.e.*, a steam-jacketed cylinder provided with a rotating helical stirrer. The steam supplied to the jacket of the "beater" operates a steam ejector by which a vacuum is produced in the "beater." H. M. LANGTON.

Distilling fatty acids and other volatile substances from oils and fats, acid oils, and crude fatty acids. J. J. V. ARMSTRONG. From NAAML. VENNOOTS. A. JURGENS' MARGARINEFABR. (E.P. 248,828, 10.12.24).—Pure odourless neutral or almost neutral undecomposed fats or oils and pure fatty acids are obtained by a distillation process charac-

terised by the following features. A very short period of heating of the liquid phase is obtained by continuously supplying the raw material and continuously and completely removing the whole residue of the distillation. A large surface of contact between the liquid and the steam or inert gases used to distil the fatty acids is obtained by causing the oil to flow in a thin layer or film over a perforated plate placed in a drum, the steam or inert gases being introduced underneath. A reduced pressure is applied in order to keep the consumption of steam or inert gases (in spite of the relatively low temperature) within economical limits. The temperature of the liquid phase measured anywhere within the liquid-flow system is not allowed to exceed 250°. H. M. Langton.

Rendering fat- and oil-containing solids of animal origin. W. Laabs, Assr. to Allbright-Nell Co. (U.S.P. 1,578,245, 23.3.26. Appl., 22.6.25).—The moisture content of the material is reduced to 60% by agitating and heating at temperatures and pressures not sufficiently high to cause substantial disintegration and separation of oils and fats. The temperature is then raised above 117° and by agitation and the action of high-pressure steam developed from the natural moisture content, the material is disintegrated and the oils and fats are liberated. When the moisture content has fallen to 17% the pressure is reduced to less than 1 atm. and agitation continued at temperatures below 117° until the moisture content is substantially under 15%, when the solid residue is pressed.
E. H. Sharples.

Distillation of oils, liquid fats, hydrocarbon mixtures, and similar liquids. C. Melhardt (G.P. 424,678, 18.1.25).—Low-boiling constituents are distilled off by forcing the liquids through water or dilute salt solution, which is kept boiling vigorously in a space between members of porous material.
L. A. Coles.

Purifying and deodorising oils and fats. Naaml. Vennoots. A. Jurgens' Vereenigde Fabr. (Austr. P. 96,154, 7.4.21).—Oils and fats are treated with steam or inert gases other than hydrogen, if necessary *in vacuo*, in the presence of inert material upon which are deposited finely-divided oxides, basic carbonates, or silicates of heavy metals, preferably with the addition of finely-divided iron or fuller's earth. L. A. Coles.

Preparation of sulphur soaps. Maschinenbau-Anstalt Humboldt (G.P. 424,499, 6.2.25).—Fatty acids, oils, or fats, alone or mixed with resins, resin acids and other substances imparting smell, or colour, or acting medicinally, are saponified with polysulphides. Soaps are formed from which the sulphur can be dissolved out by water in an active colloidal form. W. Clark.

Decomposing fats, oils, and waxes. W. Schrauth (U.S.P. 1,576,005, 9.3.26, Appl., 12.4.24).—See Can. P. 245,373; B., 1926, 287.

Cooling of liquids to form powdered or granular solids, particularly soap powder. H. Guttin (E.P. 250,441, 17.7.25).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Particle size of pigments and its influence on paints and paint films. C. P. van Hoek (Farben-Ztg., 1926, 1237—1239, 1295—1297, 1350—1353, 1399—1401, 1455—1457, 1513—1516).—A general review including the influence of particle size on the protective power, the adhesion of the particles to the medium, the colloidal character of paint suspensions, flocculation, the determination of particle size, and its dependence on the method of preparation of the material and the grinding of the paint, and the relation of particle size to the oil content, colour and covering power of paint. A bibliography is given. D. F. Twiss.

Some synthetic organic colouring matters used in lake making. F. M. Rowe (J. Oil Colour Chem. Assoc., 1926, **9**, 56—80).—A review of the typical synthetic colouring matters used for lakes, mainly with reference to their constitution, and to their physical properties that largely depend upon the constitution. The colours are considered under the headings :—Azo-, pyrazolone-, nitroso-, nitro-, quinoline-, ketonimine-, triphenylmethane-, xanthene-, thiazine- and anthraquinone-derivatives.
S. S. Woolf.

Antimony sulphides. L. M. Currie (J. Physical Chem., 1926, **30**, 205—238).—A study of the nature and number of phases in the so-called antimony pentasulphides, of the colours of the antimony sulphides, and of the factors affecting these. In agreement with Bosek (J.C.S., 1895, **67**, 519), pure antimony pentasulphide can be prepared by following the procedure of Bunsen (Annalen, 1878, **192**, 305). Cold sulphur solvents have no effect on the pure pentasulphide, but with hot solvents, especially carbon disulphide, some decomposition occurs. The passage of a slow stream of hydrogen sulphide through solutions of quinquevalent antimony reduces some of the antimony, forming the trisulphide and sulphur. Long-continued passage of the gas, high temperatures, and low acid concentration favour the formation of the trisulphide. The decomposition of sodium thioantimonate by dilute acids yields a mixed pigment consisting of sulphur and a solid solution of sulphur in antimony tetrasulphide, the amount of which depends on the conditions of decomposition. The composition of this solid solution can be varied between the limits corresponding to $Sb_2S_{4\cdot66}$ and Sb_2S_4. Rise in the temperature of reaction or of drying, the passage of a current of air or hydrogen sulphide, or precipitation in presence of free alkali, are all accompanied by an increase in the amount of free sulphur formed, as a separate phase, at the expense of that in the solid solution. Contrary to Hahn (Kolloid-Z., 1922, **31**, 200) and others, sodium thiosulphate and tervalent antimony in dilute acid give antimony trisulphide and sulphur, no pentasulphide, oxysulphide, or solid solution

being formed. The sulphur content, in this case, is increased by raising the temperature of mixing, the ratio of thiosulphate to antimony, the concentration of thiosulphate, and the acidity of the final mixture up to a limit (approx. 30%). All attempts, other than mechanical, to add sulphur to the trisulphides failed. The pure pentasulphide is more or less completely decomposed at 70—75° yielding the tetrasulphide. This, in turn, is slowly decomposed at 105° and rapidly at 120—155°. At 65° and below decomposition is slight and very slow. Intimately mixed sulphur appears to retard the decomposition of the tetrasulphide, the solid solutions of Sb_2S_4 and S being stable above 100°. At 105—108°, the rate of decomposition is slow, becoming rapid at 112—115°; at 150° decomposition is complete in a few hours. Above 150° oxidation or sublimation of the sulphur chiefly occurs, with an accompanying change in the physical properties of the trisulphide formed. *X*-Ray examination shows that the precipitated sulphides of antimony are all amorphous solids. Prolonged heating near 200° converts them into the black crystalline variety of the trisulphide. The amorphous trisulphide in a finely divided state is pale yellow; larger particles are crimson. Heating the suspensions favours coagulation and darkens the colour of any precipitated sulphide. Addition of gelatin, up to a limit, prevents agglomeration and the suspensions and precipitates become lighter in colour as a result. Acids tend to flocculate, and alkalis to peptise it, with the expected colour changes. Maroon-coloured antimony sulphides are mixtures of crimson and black sulphides. Electrical disintegration of stibnite by Svedberg's method gave a yellow crystalline trisulphide (finely-divided form) which had an -ray pattern identical with that of the natural sulphide, and with that of the artificially prepared crystalline sulphides of antimony. Antimony pigments precipitated in the presence of S or SH ions give various shades ranging from gold to brown and, finally, to black. The difference between the maroon and brown modifications is due to adsorption of hydrogen sulphyde by the latter, which, by preventing intimate contact of the particles, prevents the formation of the larger crimson particles. The effect of heat on the colour of the penta- and tetrasulphides is but slight, a general darkening only being noticeable.

L. S. THEOBALD.

U.S. Government master specification for shellac, flake orange. (U.S. Bureau of Standards, Circ. No. 302, Feb. 9, 1926, 6 pp.)—Four types, A, B, C, and D, of the manufactured product of stick lac, freed from most of the lac dye and prepared in "free" flake form are defined (seed, garnet, and button lac being excluded). Types A, B, and C must show not more than 1·75, 2·50, and 3·00%, respectively, of matter insoluble in hot 95% alcohol, other requirements being the same for the three types, viz.:—maximum for iodine value 18·0, moisture and volatile matter 2·0%, water-soluble matter 0·5%, wax 5·5%, and ash 1·0%. Type D agrees with type C except in iodine value, where the maximum is raised to 24·5%. Colour is not specified except in the use of comparison samples mutually agreed on by buyer and seller. Methods of sampling and testing are detailed.

S. S. WOOLF.

Method for estimating the colour depth or brightness of resins, varnishes, oils, etc. E. FONROBERT and F. PALLAUF (Farben-Ztg., 1926, 31, 1354; cf. B., 1926, 201).—In connexion with the method described earlier, the use of colorimeters consisting of glass prisms filled with iodine solutions of known strength is recommended; these show very little alteration on storage.

D. F. TWISS.

Drying of fatty oils. L. AUER (Farben-Ztg., 1926, 31, 1240—1241).—Linseed oil films in an atmosphere of carbon dioxide or in a vacuum (10 mm. pressure) dry in the course of a few days and show an increase in weight. This result is attributed to a primary coagulation of the oil by gas similar to that of a lyophobic sol by an electrolyte; in the high vacuum produced by a mercury pump no drying was observed. The increase in weight arises during the transfer from the containing vessel to the balance. When drying occurs in the open atmosphere, the coagulation and adsorption are concurrent; chemical combination succeeds the adsorption.

D. F. TWISS.

Drying process of the "drying oils." H. WOLFF (Farben-Ztg., 1926, 31, 1239—1240, 1457; cf. B., 1925, 45).—Linseed oil varnishes will dry slowly in an atmosphere containing so little oxygen that the drying process must be essentially of colloidal type, and is possibly independent of the oxidation reaction.

D. F. TWISS.

German oil of turpentine. I. BISCHOFF (Farben-Ztg., 1926, 31, 1517).—Analytical and practical tests with a sample of German oil of turpentine indicate this to be as good as the imported article for many applications.

D. F. TWISS.

New methods of varnishing with cellulose esters. H. RASQUIN (Farben-Ztg., 1926, 31, 1293—1294).—The advantages and disadvantages of the method of varnishing by spraying nitrocellulose are discussed and modified processes indicated enabling the cheaper application of such varnishes by means of a brush.

D. F. TWISS.

Foam meter. WILLIAMS.—See I.

Apparent viscosity of solutions of nitrocotton. MCBAIN, HARVEY, and SMITH.—See V.

Exposure tests with pointolite lamp. HOCHHEIM and KNEBEL.—See VI.

High and low stiffening carbon blacks. SPEAR and MOORE.—See XIV.

PATENTS.

Process of making carbon black. O. G. MESSENGER, Assr. to STANDARD DEVELOPMENT CO. (U.S.P. 1,577,481, 23.3.26. Appl., 18.10.23).—Hydrocarbon gases are made to react with successive

small proportions of oxygen so as to effect the liberation of carbon. D. F. TWISS.

Carriers for dyes etc. MAISON BRETON, J. FICHOT & CO. SUCCESSEURS, and G. L. A. CRUT (F.P. 599,868, 23.9.24).—As carriers there are used the precipitates obtained on treating solutions of aluminium sulphate with quantities of alkali carbonates or ammonium carbonate insufficient for complete precipitation. These products are less transparent than those obtained with larger quantities of soluble carbonate. B. FULLMAN.

Production of red antimony sulphide pigments. P. J. F. SOUVIRON (F.P. 600,894, 18.7.25).—By treating antimony solutions with thiosulphate in the presence of an aluminium salt, antimony sulphide pigments of strong colour and great stability towards light and heat are obtained. D. F. TWISS.

Manufacture of polymerised styrene and its homologues. NAUGATUCK CHEMICAL CO., Assees. of I. OSTROMISLENSKY (E.P. 233,649, 15.9.24. Conv., 7.5.24).—The patent relates to the preparation from styrene and its homologues of vitreous polymerisation products, which are tough, transparent, practically colourless if necessary, of relatively high melting point, and capable of being cut in thin films or parings, and which differ in several respects from the resinous polymerisation products of similar chemical composition. The styrene and its derivatives are obtained from the products of pyrogenetic decomposition described in E.P. 232,909 (B., 1926, 299), *e.g.*, styrene, *o*- and *p*-methylstyrene, *s*-dimethylstyrene, and ethylstyrene. In every case the compound $Ar{\cdot}CH:CH_2$, mixed with at least one other substance, is polymerised to the "resinous" polymer by heating at 140—220° (temperatures differing in different cases), the resinous polymer purified, depolymerised by heating at 350—600°, the $Ar{\cdot}CH:CH_2$ purified, and polymerised to the vitreous polymer by heating at 135—140°. For example, crude styrene (from the pyrogenetic decomposition of ethylbenzene) is heated at 140—180°, for 16—48 hrs., in an autoclave or under reflux. The resinous styrene polymer (90—100% of the original styrene) is separated by steam or heat distillation, and dry-distilled at 350—500°, the product yielding on purification 45—55% of styrene (containing a small quantity of low-boiling impurity). A 50—65% solution of this substance in xylene is heated at 135—140° under reflux, when a solution is obtained from which "vitreous" polymerised styrene, in 70% yield, may be obtained by evaporation, precipitation with alcohol, etc. In many cases the final polymerisation is carried out in the absence of a solvent. B. FULLMAN.

Manufacture of polymerised styrene. NAUGATUCK CHEMICAL CO., Assees. of I. OSTROMISLENSKY and W. A. GIBBONS (E.P. 243,768, 15.9.24. Conv., 7.5.24).—"Vitreous" polymerised styrene (cf. preceding abstract) is prepared by heating a mixture containing at least 40% of styrene (such as a solution of styrene in an organic solvent) at 135—200°, the temperature depending on the relative concentrations of styrene and the other substances. For example, a mixture of styrene (84%) and ethylbenzene is heated under reflux for 25 hrs. at 175°, and the product then purified by heating *in vacuo* (to ensure absence of air) with the temperature rising slowly to about 130°, to remove volatile residues. If high-boiling impurities be present the substance is dissolved in a low-boiling solvent (*e.g.* benzene), which is then evaporated; or the polymerised styrene in the crude product may be precipitated by addition of alcohol, solution in benzene, re-precipitation, and final evaporation as usual. Details are given of the preparation of suitable styrene mixtures, for instance, those obtained by distillation of the product obtained from the tube furnace in the pyrogenetic decomposition of ethylbenzene as described in E.P. 232,909 (B., 1926, 299). B. FULLMAN.

Artificial resins. BRIT. CYANIDES CO., LTD., and E. C. ROSSITER (E.P. 248,477, 5.12.24).—The almost universal presence of formic acid in commercial formaldehyde imparts an acid reaction to thiocarbamide-formaldehyde condensation products with possible harmful action. This is obviated without the introduction of alkaline products, by passing a current of dry air or an inert gas through the solution while it is being evaporated at a temperature below 100°. S. S. WOOLF.

Dispersion of unbleached shellac. SIEMENS & HALSKE A.-G., Assees. of C. HARRIES and W. NAGEL (G.P. 421,337, 15.2.23).—Peptising agents such as organic acids yield disperse phases, which may be again completely taken up by alcohol. Small quantities of mineral acids exert a coagulating effect. B. FULLMAN.

Linoleum cement. P. SLANSKY, and DEUTSCHE LINOLEUM-WERKE HANSA (E.P. 248,974, 5.6.25).—In the production of linoleum cement by melting solid oxidised oils with resins in an open pan at temperatures below 150—160°, oils of a limited state of oxidation only may be used, the higher temperatures required to melt more strongly oxidised oils causing ignition and crumbling. If, however, the melting is carried out in an autoclave in the absence of oxygen, the more highly oxidised oils may safely be used. The melting is accelerated by the addition of not more than 10% of water. S. S. WOOLF.

Heat-plastic materials (E.P. 249,172).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

"Cyclopentadiene rubber." A new cyclic synthetic polymerisation product. H. A. BRUSON and H. STAUDINGER (Ind. Eng. Chem., 1926, **18**, 381—383).—*cyclo*Pentadiene as a methylene derivative of butadiene should be capable of polymerisation to a "rubber." Several halogen compounds, particularly stannic chloride, act as catalysts, polymerisation being effected in chloroform at 0°. A quantitative yield is obtained of a product closely resembling freshly coagulated rubber; it readily

undergoes atmospheric oxidation to $(C_5H_6O)_x$, forms a bromine additive compound $(C_5H_6Br_2)_x$, a nitrosite, and a highly coloured hydrochloride and hydrobromide; vulcanisation with sulphur chloride gave products ranging from a soft jelly to a vulcanite-like material. D. F. TWISS.

High and low stiffening carbon blacks [in rubber or oils]. E. B. SPEAR and R. L. MOORE (Ind. Eng. Chem., 1926, 18, 418—420).—No simple relation exists between the stiffening power of carbon blacks and their adsorptive power for organic dyes or hexamethylenetetramine. High tensile strength in a rubber mixing containing a carbon black is always associated with the colloidal dispersion of at least a portion of the carbon in the rubber matrix, the colloidal particles being too small to be resolved by the microscope; the stiffening effect, however, appears to be independent of this feature. Mere particle size is undoubtedly a factor but not to the exclusion of other influencing conditions. D. F. TWISS.

Internal mixers for rubber stocks. P. S. SHOAFF (Ind. Eng. Chem., 1926, 18, 345—347).—A review of the advantages and disadvantages of internal mixing machines relative to roll mills. Recommendations are made as to the order of procedure for mixing various types of rubber "compounds." D. F. TWISS.

Vulcanisation in solution. W. GREINERT and J. BEHRE (Kautschuk, March, 1926, 63—64).—The quality of unworked rubber is indicated by the period necessary for gelation when 10 c.c. of a solution of the dry rubber in dry benzol is treated with 1 c.c. of an 11% solution of sulphur chloride in benzol (cf. Le Blanc and Kröger, B., 1921, 667 A; 1924, 141). The method not only distinguishes between low-grade and high-class forms of rubber but reveals marked differences between various samples of pale crêpe rubber. The adverse effect of mastication is evident not only in the prolongation of the gelation period, but also in the behaviour on hot vulcanisation with sulphur; in this case the resistance to load is decreased with consequent modification of the stress–strain curve. D. F. TWISS.

Accelerated ageing tests [for rubber]. C. R. PARK (Rubber Age, London, 1926, 7, 64—68; Kautschuk, March, 1926, 57—61).—The requirements for an accelerated ageing test for rubber are that a given period shall be equal to a definite period of natural ageing and that the physical properties shall change in the same direction and to a comparable degree in accelerated and in natural ageing. The behaviour of vulcanised samples containing rubber and sulphur only, or rubber, sulphur, and zinc oxide with mercaptobenzothiazole, hexamethylenetetramine, diphenylguanidine, and litharge respectively reveals no simple relation between the alteration in the stiffness index and in the tensile product on ageing naturally, at a higher temperature, and at a higher temperature by oxygen under pressure, respectively. Natural ageing is probably a more complex phenomenon than has been assumed hitherto and the inadequacy of accelerated ageing tests is attributable to over-emphasis or neglect of certain factors. D. F. TWISS.

Oxidation of rubber exposed to light. I. WILLIAMS (Ind. Eng. Chem., 1926, 18, 367—369).—Natural oxidation of vulcanised rubber may result in general deterioration throughout, in formation of a surface film, or in the development of cracks. Exposure to light aids surface oxidation with formation of an almost inelastic film, light of shorter wave length being more active. Surface cracking or "checking" under the influence of ultra-violet light will not occur in the absence of air and is attributable to the formation of ozone; unstrained rubber is not "checked" by ozone. Surface oxidation hinders the development of "checking," which is consequently retarded by the surface application of a copper salt. D. F. TWISS.

Addition of light to accelerated ageing [of rubber]. F. P. JECUSCO (Ind. Eng. Chem., 1926, 18, 420—424).—Exposure to light during accelerated ageing of vulcanised rubber at 71° expedites the decrease in strength, and the increase in the acetone extract; the rubber undergoes a smaller loss in weight than when not illuminated and becomes covered with a red insoluble coating. This activity is not restricted to ultra-violet light; green and infra-red radiation also have a marked effect, especially on rubber activated by previous ageing (*cf.* Asano, B., 1925, 816). D. F. TWISS.

New mechanical test for rubber insulation. C. L. HIPPENSTEEL (Ind. Eng. Chem., 1926, 18, 409—411).—A 2-inch length of insulated wire, without braid, is compressed between two parallel steel surfaces which approach one another at a uniform rate. A small self-recording machine is described, the record of the compression of the insulation forming a sort of stress-strain curve. D. F. TWISS.

Economical use of reclaimed rubber as a substitute for new rubber. J. M. BIERER and C. C. DAVIS (Indiarubber J., 1926, 71, 565—571; Ind. Eng. Chem., 1926, 18, 348—353; cf. Holt and Wormeley, U.S. Bur. of Standards Tech. Paper 294).—It is possible to replace part of the new rubber, even in a tyre tread, by a correspondingly greater amount of suitable reclaimed rubber without loss of abrasion resistance or depreciation of the stress-strain curve; the procedure roughly is a replacement of new rubber by approximately the same volume of rubber component in the reclaimed rubber, with a simultaneous adjustment of the quality, necessitated by the lower value of the old rubber component, by alteration of the other compounding ingredients. It is desirable to maintain a high proportion of carbon black. Depreciation of ageing qualities due to the use of reclaimed rubber from inner tubes can be effectively obviated by the introduction of suitable anti-oxidants. Selenium greatly increases the abrasion-resistance of rubber. D. F. TWISS.

Patents.

Heat-plastic materials [from rubber]. E. A. White. From B. F. Goodrich Co. (E.P. 249,172, 24.9.24).—Well-masticated rubber (100 pts.), if necessary with the addition of pine tar, is mixed with an isomerising agent (up to 15 pts.), containing the grouping $R \cdot SO_2X$, *e.g.*, sulphuric acid, an organic sulphonic acid or a sulphonyl chloride, or with a mixture of such agents. The mixture is then heated between 110° and 175°, and reworked after cooling. According to details of the treatment, the product may be tough and heat-plastic like balata, or hard and friable, but yielding on fusion a mass resembling, and capable of replacing, shellac. D. F. Twiss.

Accelerator for vulcanisation of rubber. P. M. Paulson, Assr. to Roessler and Hasslacher Chemical Co. (U.S.P. 1,575,865, 9.3.26. Appl., 17.6.25).—Vulcanisation of rubber is expedited by the addition of a compound of the formula $NH_2 \cdot C(:NH) \cdot NRR^1$, where R and R^1 are dissimilar organic radicals. D. F. Twiss.

Accelerator for vulcanisation of rubber. C. S. Williams, Assr. to Roessler and Hasslacher Chemical Co. (U.S.P. 1,575,884, 9.3.26. Appl., 17.6.25).—Rubber is vulcanised with the aid of an accelerator of the constitution $NH_2 \cdot CH(:NH) \cdot NHR$, where R is a hydrocarbon radical. D. F. Twiss.

Vulcanising rubber. N. A. Shepard and S. Krall, Assrs. to Firestone Tire and Rubber Co. (U.S.P. 1,576,072, 9.3.26. Appl., 20.6.23. Renewed 4.8.25).—A mixture of rubber with a small amount of a reaction product of an aldehyde-ammonia and a mustard oil is heated with a vulcanising agent. D. F. Twiss.

Vulcanising caoutchouc. D. N. Shaw, Assr. to Goodyear Tire and Rubber Co. (U.S.P. 1,580,089, 6.4.26. Appl., 18.11.24).—Rubber is heated with a vulcanising agent, a nitrosocarbazole, and an activator. D. F. Twiss.

Vulcanisation of rubber. Atlantic Dyestuff Co., Assees. of A. C. Burrage, jun. (Can. P. [a] 245,929, 27.2.24, [b] 245,930, 27.2.24, and [c] 247,108, 19.2.23).—(a) Diphenyl-*o*-tolylguanidine is used to accelerate vulcanisation. (b) Compounds of the type of diphenyl-*o*-tolylguanidine for use as vulcanisation accelerators are produced by the action of litharge and an aromatic amine on a diarylthiocarbamide. (c) In vulcanising rubber, a mixture of accelerators, *e.g.*, of arylthiocarbamides with other accelerators or of methylene-*p*-toluidine with tri-*o*-tolylguanidine, is used, the constituents becoming active at different temperatures. D. F. Twiss.

Separation of textile from rubber in rubber goods. P. Marmier and F. de Gaalon (F.P. 598,722, 14.5.25).—The materials (tyres) are heated in a closed vessel under pressure with a mixture of 3 pts. of benzine and 1 pt. of vaseline oil, in the presence of turnings of copper, tin, or lead. B. Fullman.

Regeneration of vulcanised rubber. L. Gallois (F.P. 599,869, 23.9.24).—Vulcanised rubber is heated in a closed vessel under pressure with rubber solvents, *e.g.*, benzine, benzol, petroleum, xylene, or turpentine oil, to about 150—200°. On evaporation of the solvent, a vulcanisable regenerate is obtained. B. Fullman.

Hydrogenation of artificial and natural rubber. Siemens u. Halske A.-G. (G.P. 424,281, 24.2.23). —The rubber, after being made sufficiently plastic, is dissolved in an organic solvent and treated with hydrogen, in the presence of metallic catalysts. B. Fullman.

Production of rubber articles. R. Ditmar (Austr. P. 99,664, 27.11.23).—Rubber latex is mixed with 0·1—10% of glycerol as an anti-coagulant, and with compounding ingredients, including sulphur; it is then dried, moulded, and vulcanised. Fibrous or porous materials, *e.g.*, fabrics or bricks can be impregnated with the compounded latex by spraying. D. F. Twiss.

Rubber vulcanisation. Dovan Chemical Corp. (E.P. 226,836, 29.12.24. Conv., 29.12.23).—See U.S.P. 1,546,876; B., 1925, 1000.

XV.—LEATHER; GLUE.

Chemico-histological study of leather manufacture. I. Structure and properties of freshly flayed steer skin. H. G. Turley (J. Amer. Leather Chem. Assoc., 1926, **21**, 117—156).—The routine histological procedure used is described. By the use of simple stains, the skin proteins are classified into acidic and basic. The following elements are described in detail:—*stratum corneum*, *stratum granulosum*, *stratum germinativum*, hyaline layer, *corium minor*, hair and hair root sheaths, sebaceous glands, sudoriferous glands, muscle, wandering cells, elastic fibres, blood vessels and lymphatics, grain membrane, *corium major*, connective tissue fibre bundles, aerolar tissue sheaths, and the interfibrous material. On staining certain sections with thionin or toluidine-blue, and Bielschowsky stain respectively, the hyaline layer was shown to be different from collagen. It resembles the epithelial tissue of the epidermis and seems to be identical with the basement membrane of histologists. The elastic fibres are present mainly in the grain layer, and only to a small extent elsewhere. The elastic fibres in the grain layer are 2 μ in diameter and 1 μ or less in the grain network. They are basic, *i.e.*, stain with acid dyes. The connective tissue fibres in the grain layer are 5·20 μ wide. They are studded with nuclei of collagen-forming cells. The nuclei are large, flat, oval discs about 16 $\mu \times 5$ μ, and since they are seen on edge and at various angles it is inferred that there is no general symmetrical arrangement of the collagen fibres. The large bundles of connective tissue fibres are 180 $\times$ 100 μ, and somewhat elliptical in cross section, and the individual fibres 55 $\times$ 23 μ. The connective tissue fibrillæ are 3—6 μ

in diameter. The findings are illustrated by photomicrographs and a coloured plate made from stained sections. D. WOODROFFE.

Action of lime on enzymes. R. L. COLLETT (J. Soc. Leather Trades Chem., 1926, 10, 100—112).—Pancreatic trypsin, bacterial proteases from *Staphylococcus albus*, pancreatic lipase, "thrombase," urease, and yeast amidase respectively were mixed with milk of lime and at definite intervals, the activity of the enzyme was measured. The proteolytic and fat-splitting enzymes were rapidly inactivated by the lime. "Thrombase" was inactivated, and did not function at the alkalinity of tannery lime liquors. There was no evidence that the thrombase system had a secondary proteolytic stage which might suggest that it affected unhairing. These results and those described previously (B., 1924, 25) show that neither bacteria nor enzymes, by acting in lime liquors, can produce the ammonia which is always found in old or mellow limes. Lime itself, however, acting on digested proteins produces large quantities of ammonia. D. WOODROFFE.

Official method [of Society of Leather Trades Chemists] of quantitative tannin analyses (J. Soc. Leather Trades Chem., 1926, **10**, 30—42).—The method is essentially the same as that outlined in the Leather Chemists' Pocket Book (Spon, 1914). Evaporating basins must be shallow, flat-bottomed, without sharp angles, and not less than 6·5 cm. in diameter. Porcelain basins may be used, but silver ones are preferable. Residues shall be dried in a water, steam, vacuum steam, or electric oven at a uniform and constant temperature of 98·5—100°. Berkefeld filter candles shall be used for the filtration of the tannin infusions. Distilled water must be free from sulphates and chlorides, have p_H 5·0—6·0, and leave a residue $<$ 0·002 g. per 100 c.c. Kaolin should be washed with hydrochloric acid and then with distilled water; 1 g. suspended in 100 c.c. of water should show p_H 4·0—6·0. The residue from 100 c.c. of 0·01 *N*-acetic acid shaken with 1 g. of kaolin should be less than 1 mg. Tanning materials must be ground to pass through a sieve of 5 wires per cm. Fibrous materials are to be ground, separated into fine and coarse portions, each weighed, and the same proportions of each used together for analysis. Samples of ground sumach must be thoroughly mixed in a rotating churn before weighing out for extraction. Solid extracts shall be ground in a mortar before weighing. Pasty extracts or extracts of uneven moisture content are to be weighed in a flat-bottomed basin, dried in an air oven at 70°, exposed overnight to the laboratory atmosphere, weighed, and loss of water calculated. The extract is then powdered and a weighed portion dried in the oven at 98·5—100°. The total loss in moisture is calculated. Liquid extracts should be mixed in a shaking machine. Viscous extracts should be heated to 45° on the water bath, well mixed, cooled, and then weighed out at once. The tannin infusion must contain 3·75—4·25 g. of tannin per litre. Liquid extracts must be washed with boiling distilled water into a litre flask. Sumach, myrobalans, and synthetic tanning extracts must be diluted with water at 70°. Solid and pasty extracts shall be heated on the water bath with ten times their weight of water until no solid fragments remain, except fibrous matter. Solid tanning materials shall be extracted in a Procter extractor. The material must be soaked overnight in cold distilled water, the cold infusion ($<$ 100 c.c.) siphoned off, the water bath heated to 45—50°, 100 c.c. of distilled water at 45—50° poured on to the material, left for 20 min., and then siphoned off rapidly. This is to be followed by three similar macerations, after which the temperature of the bath is raised to boiling, and four macerations with 125 c.c. of boiling water carried through. The infusion must be well mixed and cooled to 17—18° by immersing the flask in a large vessel containing water. The cool infusion must be made up to 1 litre, well mixed, and filtered at once. The moisture in solid tanning materials and solid and pasty extracts shall be determined directly on the material, using a squat-shaped, wide-mouthed weighing bottle. Infusions must be filtered, repeatedly if necessary, until "optically clear," both by reflected and transmitted light. The non-tannins are determined by shaking 100 c.c. of the infusion with a known weight of chromed hide powder for 15 min. Specific gravity must be determined by the pyknometer or bottle at 15°. Four samples of materials should be taken, packed in glass bottles, and sealed. In a consignment containing x packages, $0·7 \times \sqrt{x}$ packages shall be sampled, the samples taken shall be mixed together, and the bulk reduced by quartering to the desired size. Detailed instructions are given for sampling the different kinds of materials. The analysis of used liquors and spent tanning materials is performed in the same way. Used liquors must be diluted to *d* 1·007—1·008. For weaker liquors the amount of dry hide powder used in the detannisation shall be 1 g. for every 0·001 the *d* is greater than 1·000. The amount of spent tanning materials taken for analysis shall be sufficient to yield an infusion containing 3·5—4·5 g. of tannin per litre if possible, but in no case must the total solids exceed 10 g./litre. Analytical results for spent tan must be calculated on the dry material and for spent or used tan liquors be reported in g./100 c.c. The density of used liquors must be reported. D. WOODROFFE.

Sampling tanning materials, leather, etc. for analysis. H. G. BENNETT (J. Soc. Leather Trades Chem., 1926, 10, 57—58).—By a slight modification of the formula $n=0·7\sqrt{x}$ for sampling (*cf.* preceding abstract) it becomes $x = 2n^2$, where x is the number of packages and n the number to be selected for sampling. D. WOODROFFE.

Report of a committee of the French section of the Society of Leather Trades Chemists on quantitative tannin analysis. G. HUGONIN (J. Soc. Leather Trades Chem., 1926, 10, 89—100).—Comparative analyses by different chemists have been made on various liquid tanning extracts using the official chromed hide powder and powders

chromed with other solutions. A powder chromed with a 10% or 12·5% solution of normal chromium chloride gave lower figures for non-tannin than the others. Normal chrome alum solutions gave high non-tannin figures, due to less dissociation by the chromium sulphate. Determinations of the p_H value by electrometric and colorimetric methods showed a lower value by the latter method. The electrometric method yielded more concordant results by different observers. Solutions detannised with powder chromed with normal chromium chloride yield non-tan liquors with a lower p_H value than those detannised with the other powders. Comparison of the Berkefeld filter candle with centrifuging and sedimentation shows that it gives a higher content of insoluble matter. Particles of 1 μ or larger should be classed as "insoluble matter." The chroming of hide powder with normal chromium chloride is favoured. D. WOODROFFE.

Behaviour of synthetic tanning materials towards hide substance. E. WOLESENSKY (Tech. Papers, U.S. Bur. Standards, 1926, 20, [309], 275—287).—A study has been made of the hydrolytic action of synthetic tannins on hide substance during tanning, the rate of fixation of the tanning material by the hide, and the maximum amount of tanning material which may be fixed under various conditions of concentration and length of treatment. The hydrolytic action on the hide varied with the tanning materials used, being nil for one derived from cresol; 3—6% of hide substance was dissolved by using synthetic tannins derived from phenolsulphonic acid, and from cresol and sulphuric acid, also a commercial product of unknown composition. Synthetic tanning materials produced by condensing a sulphonated aromatic compound with formaldehyde combine rapidly with pelt but not in large quantity. Those produced by sulphonating the product of condensation of an aromatic compound with formaldehyde, possess greater filling power, and require longer time to reach maximum absorption by the pelt; their absorption depends on the concentration. Synthetic tanning materials of the former group appear to combine chemically only. Both chemical and colloidal combination enter into the absorption of the latter group of synthetic tannins by pelt. High concentration and long treatment with synthetic tannins are unnecessary, especially with the former group, and may be distinctly harmful.

D. WOODROFFE.

Polluted atmosphere a factor in the deterioration of bookbinding leather. F. P. VEITCH, R. W. FREY, and L. R. LEINBACH (J. Amer. Leather Chem. Assoc., 1926, 21, 156—176; cf. B., 1901, 819).—Leather from the back and sides of various old leather-bound volumes has been analysed. The leather in the binding most exposed to air and light was most deteriorated, and contained in the water extract the highest amounts of free mineral acid, of sulphates, and of nitrogen as determined by distillation with magnesia. The direct correlation between deterioration, chemical composition, and degree of exposure has been confirmed by comparative analyses of leather from the sides, backs, top edges, and bottom edges of books, portions covered with skiver and uncovered portions, embossed and unembossed leathers. The increase in sulphur or sulphate content of leather during exposure must result from an absorption of those components from the air, and since bindings that had not been exposed to gas lighting were not exceptional, the deterioration resulting from exposure is attributed to the absorption of sulphurous and acidic impurities from the air of large towns. The nitrogen content shows that the deterioration of the leather is partly the result of a slow hydrolysis of the hide substance by the acid originally present and that taken up from the air. Short-life bindings had a high acidity (4·80—9·10%; 0·68—3·67% in the sides; determined by the Procter–Searle method), indicating the presence originally in the leather of excessive quantities of acid. Several very old bindings (91—161 yrs.) had a low acidity in the sides (–1·6 to 2·8%) and some had only 0·1—0·5% of free acid in the back. Slight alkalinity or very low original acidity promotes longer life in leather bindings. Coatings, finishes, oils, greases, and waxes are beneficial as they prevent the absorption of atmospheric impurities.

D. WOODROFFE.

Acetylation of tannin. FERNÁNDEZ and FERNÁNDEZ.—See XX.

PATENTS.

Preparation of tanning materials. BADISCHE ANILIN- & SODA-FABR. (G.P. 420,593, 8.2.24).—The materials are prepared from substances of a coaly nature, fossilised or of recent origin, by the action first of dilute and then of stronger nitric acid. For example, finely powdered wood charcoal is boiled under reflux with 40% nitric acid for 6 hrs., and the paste obtained on filtration is slowly brought to boiling in 96% acid and boiled for two days. The solution yields on evaporation an orange-yellow soluble tanning material, applicable at once or after partial neutralisation. Humus coal, peat, and lignite also yield tanning materials on similar treatment.

B. FULLMAN.

Process for tanning hides. BADISCHE ANILIN- & SODA-FABR., Assees. of A. MITTASCH and O. BALZ (G.P. 420,645, 4.12.21).—The tanning is carried out by means of aqueous solutions of products obtained by the action of nitric acid on humic acid or substances rich in it, alone, or in the presence of other natural or synthetic tanning materials. The products are obtained by the action of nitric acid on substances obtained from humus coal freed from iron and bitumen. For example, humic acid or ammonium humate from humus coal is added to 65% nitric acid, and heated for some time at 70—80° with powerful stirring. The excess acid is distilled off, and a reddish-brown amorphous powder is obtained on purification by solution in water and evaporation. B. FULLMAN.

Process for tanning hides. BADISCHE ANILIN- & SODA-FABR., Assees. of K. H. MEYER and H. SCHÜTTE (G.P. 420,646, 12.5.23).—Tanning may be

effected by means of the soluble products obtained by the action of concentrated sulphuric acid or its monohydrate on crude or purified mineral oil or mineral oil fractions. For example, machine oil is stirred with sulphuric acid monohydrate for a long time at a moderately high temperature. The sulphonated product is used in moderately acid dilute solution. Similar products may be obtained from petroleum, even when free from unsaturated hydrocarbons. B. FULLMAN.

Tanning materials. BADISCHE ANILIN- & SODA-FABR., Assees. of M. EGNER (G.P. 420,647, 5.2.24).—Tanning materials are formed by mixing the formaldehyde condensation products of naphthalenesulphonic acids or their salts with the substances obtained by the oxidation, with nitric acid or nitrous fumes, of fossilised vegetable matter, *e.g.*, lignite, peat, or coal. The formaldehyde–naphthalenesulphonic acid condensation products serve the purpose of rendering the colour of the leather lighter. For example, the products obtained by the action of 40% nitric acid on humic acid or peat, or of 65% acid on lignite, are dried, and mixed with the formaldehyde–naphthalenesulphonic acid condensation product obtained in the presence of sodium hydroxide. B. FULLMAN.

Process for tanning hides. BADISCHE ANILIN- & SODA-FABR., Assees. of R. ALLES (G.P. 420,648, 14.5.24).—Pelts are treated simultaneously or successively with solutions of tanning materials obtained by the oxidation (with nitric acid or nitrous fumes) of fossilised plant substances, *e.g.*, lignite, peat, or coal, and with quebracho extract or other pyrocatechol tannins, such as pine bark extract. The use of pyrocatechol tannins prevents the production of dark-coloured leather. The leathers may afterwards be tanned by pyrogallol tannins. For example, "Casseler Braun" (a lignite product) gives on oxidation with nitric acid a soluble tannin, which is mixed with sulphited quebracho extract or gambier extract and diluted, yielding a solution which tans a delimed pelt in 6—8 days, yielding a brown, full leather. The latter, tanned moist with oak wood extract of d 1·03 for 2 days in a fulling cylinder, yields a brown, somewhat lighter coloured leather. B. FULLMAN.

Tanning materials from sulphite-cellulose waste liquors. M. HÖNIG and W. FUCHS (Austr. P. 88,650, 10.4.20; G.P. 420,802, 17.8.21).—The waste liquor is neutralised with calcium hydroxide, and boiled with 1% of the hydroxide for 1 hr. The filtered liquid is concentrated to d 1·16—1·20, and treated with an acid forming an insoluble calcium salt. A substance having the properties of a tannin is obtained. By treating the aqueous solution of the calcium salts with sulphuric acid of d 1·84 to expel volatile organic acids and then with aluminium or chromic sulphate or chromic fluoride soluble aluminium or chromium salts of the ligninsulphonic acids are formed. The filtered liquid is concentrated *in vacuo* to d 1·26—1·285. These salts do not swell pelts as much as the free ligninsulphonic acids, and give a more compact leather. B. FULLMAN.

Manufacture of gelatin. A. H. TOD (E.P. 249,736, 8.8.25).—Discoloration and deterioration of bone gelatin are due to the presence in the bone material of porous fragments which absorb and retain blood, dirt, etc. Such porous fragments are separated mechanically by means of a frame consisting of a ring, which fits closely within a vat, and is connected with a central stem by a series of radial rods on each of which is hinged a finely perforated flap adapted to close by gravity but held open by a catch rod. The bone material is immersed in a bleaching and disinfecting fluid of $d<1·116$ in the vat, the frame with the flaps open is submerged in the liquid, the catch rods are then released so that the flaps close, and the frame is raised. The porous fragments float to the surface of the liquid and are removed by the separator. D. WOODROFFE.

Electro-osmotic purification of glue and gelatin. W. RUPPEL and K. WOLF, Assrs. to AMER. ELECTRO-OSMOSIS CORP. (U.S.P. 1,577,660, 23.3.26. Appl., 13.3.24).—Glue, gelatin, and similar materials are purified by submission, in aqueous solution, to an electric current; the solution is separated from the electrodes by an anodic diaphragm of wool fabric impregnated with chromated gelatin and a cathodic diaphragm of parchment. D. F. TWISS.

Production of artificial horn. F. SCHMIDT (E.P. 230,025, 26.1.25. Conv., 27.2.24).—Mixtures containing albuminous material are gelatinised by kneading with water admixed with a volatile, water-soluble solvent, such as alcohol, acetone, or ethyl lactate, capable of dissolving cellulose derivatives and not capable of acting alone as a gelatinising agent for albumins but capable of acting as such when admixed with water. Cellulose derivatives are separately kneaded with gelatinising agents containing the same volatile solvent, or a mixture of it with water, or water alone, and the two masses are then incorporated by kneading. For example, two mixtures, one containing 30 kg. of nitrocellulose, or a mixture of it with camphor or with camphor substitute, 10 kg. of glycol diacetate, and 20 kg. of alcohol, and the other containing 60 kg. of casein, 5 kg. of glycol diacetate, 5 kg. of alcohol, and 10 kg. of water are kneaded together to a homogeneous mass in an open kneading machine, followed, if necessary by compression or rolling. L. A. COLES.

Extracting oil etc. from whale, whale blubber and flesh, fish products, etc. (E.P. 232,601).—See XII.

Pyrrole etc. from animal waste (U.S.P. 1,572,552).—See XX.

XVI.—AGRICULTURE.

Determination of available phosphoric acid of calcareous soils. S. DAS (Mem. Dep. Agric. India, 1926, 8, 69—104).—The use of 1% citric acid as a solvent for available phosphoric acid in

soils, when applied to calcareous soils, amounts, in effect, to extraction with solutions which vary in composition according to the amount of calcium carbonate present. Addition of more citric acid to neutralise the carbonate gives unreliable results. Experiments with ammonia and various salt solutions (1% ammonium citrate, sodium sulphate, ammonium sulphate, ammonium carbonate, and others) as solvents for available phosphoric acid are recorded. It was eventually found that the use of a 1% solution of potassium carbonate gave reliable results, agreeing well with expectation in the case of soils of known history and treatment. It is recommended that, for the determination of available phosphoric acid in calcareous soils, 100 g. of soil should be shaken for 24 hrs. with 1 litre of 1% potassium carbonate solution and the phosphoric acid in the extract determined by the ammonium molybdate method.

C. T. GIMINGHAM.

Significance of soil reaction in practical agriculture. M. TRENEL (Z. Pflanz. Düng., 1926, **B5**, 169—181; cf. B., 1925, 858).—Examples are given of the practical value of detailed "reaction maps" of agricultural land, which should include information as to the reaction of the subsoils. Determinations of the p_H values by the electrometric method on soil suspensions in potassium chloride solution give better correlation with crop growth than measurements in water suspensions. The soil reaction limits of various crops are given.

C. T. GIMINGHAM.

Colloidal behaviour of soils and soil fertility. II. The soil complex capable of base exchange and soil acidity. J. S. JOFFE and H. C. MCLEAN (Soil Sci., 1926, **21**, 181—195; cf. B., 1925, 858).—The problem of soil acidity in relation to base exchange is discussed. It is considered to be a question of the state of saturation or unsaturation of the soil complex capable of base exchange. The titratable acid liberated by *N*-barium chloride and potassium chloride respectively was determined for a number of soils of different origin and manurial history. The speed of cation replacement is affected by the colloidal nature of the soil and by the concentration of replaceable cations. Replacement reactions may be slowed down by temperature changes which produce coagulation. The time-rate curves are typical of adsorption reactions.

G. W. ROBINSON.

Residual effects of neutral salt treatments on the soil reaction. C. H. SPURWAY and R. H. AUSTIN (Soil Sci., 1926, **21**, 71—74).—Soils from varying depths were treated with neutral solutions of metal chlorides and after leaching out soluble products, the residual soil reaction was determined. The p_H values of the treated soils were increased, the order of effectiveness of the cations being calcium, magnesium, potassium, and sodium. Calcium chloride caused only very slight changes in reaction. Increased p_H values are ascribed to the increased solubility and hydrolysis of the soil material containing the fixed cation.

A. G. POLLARD.

Use of the quinhydrone electrode for measuring the hydrogen-ion concentration of soils. L. D. BAVER (Soil Sci., 1926, **21**, 167—179).—The saturated calomel cell is the most convenient for use with the quinhydrone electrode. The quinhydrone electrode gives results which agree closely with those obtained by the hydrogen electrode; with soils below p_H 8·0, the potential rapidly attains constancy. For accurate results 0·05 g. of quinhydrone per 15 c.c. of solution is a suitable concentration. The most desirable soil to water ratio is 1:1.

G. W. ROBINSON.

Production and use of sulphate in humid and arid soils as affected by cropping and sulphur treatments. W. W. JOHNSTON (Soil Sci., 1926, **21**, 233—244).—The formation of sulphate from sulphur applied to the soil is retarded in arid soils by growing crops which respond to sulphur treatment. In the case of humid soils crop growth increases the production of sulphate. The sulphate supply is generally sufficient under humid conditions owing to the greater ability of crops to obtain sulphur consequent on the higher hydrogen-ion concentration of humid soils. Such soils may recuperate more readily than arid soils in their sulphate content. This may explain the marked effect sometimes obtained by application of sulphur to semi-arid soils which have grown lucerne continuously for a long period.

G. W. ROBINSON.

Influence of available nitrogen on the fermentation of cellulose in the soil. J. A. ANDERSON (Soil Sci., 1926, **21**, 115—126).—The decomposition of cellulose in the soil increases with the supply of available nitrogen up to the amount of available nitrogen necessary for maximum growth of microorganisms, which, in the case of the experiments described, corresponds with a ratio of nitrogen to cellulose equal to 1:35. The ratio of cellulose fermented to nitrogen utilised decreases with increasing supply of nitrogen. Although nitrification can proceed in the presence of cellulose the nitrate produced is immediately utilised unless present in amounts more than sufficient for the requirements of bacteria. Ammonia is available for cellulose bacteria without being first converted into nitrates. Addition of carbohydrates to soil does not result in an inhibition of the soil processes. Crop failures are due to the competition of cellulose bacteria with ordinary crops for the supply of available nitrogen. Urea, aspartic acid, asparagine, alanine, tyrosine, peptone, casein, and the nitrogenous compounds of stable manure can also serve as sources of nitrogen for cellulose bacteria. In the case of the more complex compounds their availability would appear to depend on the rate at which they are hydrolysed to the amino-acid stage.

G. W. ROBINSON.

Present position and previous experience in regard to fermented manure preparations. J. WEIGERT (Z. Pflanz. Düng., 1926, **B5**, 145—161). **So-called hot fermentation of farmyard manure.** O. LEMMERMANN (*ibid.*, 162—168).—The results of

a number of experiments with fermented manure preparations and the changes taking place during the fermentation are discussed, with special reference to the material known as "*Edelmist*." The agricultural value of this manure has not yet been accurately determined on account of the difficulty of obtaining a satisfactory basis of comparison with ordinary farmyard manure, but it is concluded that such preparations are well worth further investigation.
C. T. GIMINGHAM.

Availability of nitrogen in garbage tankage and urea in comparison with standard materials. A. L. PRINCE and H. W. WINSOR (Soil Sci., 1926, 21, 59—68).—Pot experiments with barley, rape, and sorghum indicated that the fertiliser value of garbage tankage was very low. The whole of the nitrogen present, although small, becomes very slowly available. The chief value of the material is probably as a filler. In all cases urea was found to give better crop yields than ammonium sulphate and very nearly equalled sodium nitrate. The relative availability of these fertilisers was sodium nitrate, 100; ammonium sulphate, 88·2; standard tankage, 53·4; fish, 49·2; and garbage tankage, 14·2. Chemical processes for comparing the availability of nitrogenous fertilisers proved of little value. The rate of conversion of urea to ammonia was investigated in soil and sand mixtures. In soil 90% conversion occurred in 5 days. Acid soils retarded the rate of conversion, and in these cases applications of lime brought about an acceleration, proportional to the amount of lime added. A. G. POLLARD.

Potassium ferrocyanide and ferric ferrocyanide as sources of iron for plants. C. G. DEUBER (Soil Sci., 1926, 21, 23—26).—*Spirodela polyrhiza* and soya bean plants made fair growth in culture solutions containing 0·033 and 0·066 pt. per million of iron supplied as potassium ferrocyanide. Toxic effects were noted when larger concentrations of this salt were used. Ferric ferrocyanide was used satisfactorily as a source of iron for soya beans in solutions having a reaction of p_H 5·0. Growth in solutions of less acidity was unsatisfactory.
A. G. POLLARD.

Deleterious action of smoke gases on vegetation in the light of metabolism pathology. K. NOACK (Z. angew. Chem., 1926, 39, 302—304).—A series of experiments on the action of sulphur dioxide, with and without the assistance of light, on the growth of the moss, *Fontinalis*, gave results which appear to indicate that sulphur dioxide arrests the assimilation of carbon dioxide by the plant, and the photo-oxidative energy of the chlorophyll, which finds its normal acceptor in carbon dioxide, is diverted to other acceptors present in the protoplasm and in the chlorophyll itself, so that the cells are rapidly killed and the chlorophyll is bleached. A. R. POWELL.

PATENTS.

Arsenic preparations. [Insecticides and fungicides.] J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 248,975, 10.6.25).—Solutions of the yellow modification of arsenic are prepared by passing the vapours of a suitable solvent (*e.g.*, carbon disulphide or benzene) over arsenic heated in a tube. The condensate contains yellow arsenic in the form of a solution which is stable in daylight and at ordinary temperatures. Insecticides and fungicides are prepared by emulsifying the arsenic solution with solutions of soap or saponin or Turkey-red oil. A. G. POLLARD.

Preparation of insecticides. W. K. SCHWEITZER, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,574,118, 23.2.26. Appl., 29.9.24).—Dimanganous arsenate, $MnAsO_4$, prepared by heating a mixture of manganese dioxide and arsenious oxide with water at 100°, contains about 3% of soluble arsenic oxide. This can be reduced by heating the product for 18 hrs. with water and about 5% of a compound which forms with dimanganous arsenate a trimanganous arsenate. Suitable compounds are the neutral or slightly alkaline carbonates of metals which form insoluble arsenates, *e.g.* magnesium, calcium, or manganese. When manganese carbonate is used the product has the formula $Mn_3(AsO_4)_2$ and the content of soluble arsenic oxide is reduced to 1—1·5%. Trimanganous arsenate is a white powder which is of value as an insecticide. T. S. WHEELER.

Making a calcium nitrate fertiliser. BADISCHE ANILIN- & SODA-FABR., Assees. of W. WILD and K. EYER (U.S.P. 1,579,989, 6.4.26, Appl., 29.6.25).—See E.P. 246,377; B., 1926, 293.

Arsenates (U.S.P. 1,578,150).—See VII.

Di- and poly-piperidyls (G.P. 420,445).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Small-range refractometer. H. SCHULTZ (Chem.-Ztg., 1926, 50, 265—266).—The optical arrangement is similar to that of the Goerz sugar-refractometer and the range is from 1·33299 to 1·39214 corresponding with the refractive indices of pure water and a 36% sugar solution respectively. With suitable temperature control, results having an accuracy ten times greater than those given by the ordinary sugar-refractometer are obtained. E. H. SHARPLES.

Application of Lewis filtration equation. WEBER and HERSHEY.—See I.

Behaviour of starches towards dyes. HUEBNER and VENKATARAMAN.—See VI.

PATENTS.

Production of maltose from starchy materials. H. C. GORE, and FLEISCHMANN Co. (E.P. 249,428, 22.12.24. Addn. to 226,812; B., 1925, 733).—An aqueous starchy suspension, adjusted to a hydrogen-ion concentration corresponding to p_H 4·6—6, is saccharified by the addition of about 5% of malt or other diastatic agent, and maintained at 45—55° for 5—10 days in the presence of toluene as preservative. After filtering, the liquid is concentrated to a syrup, inoculated with maltose crystals, and allowed to solidify to a fondant-like mass, which is subdivided, cured by subjecting it to a suitable temperature, *e.g.*,

50°, until the particles become dry, and finally comminuted to a dry powder. C. RANKEN.

Clarifying saccharine liquors. I. H. MORSE (U.S.P. 1,573,733, 16.2.26. Appl., 29.6.25).—Cane sugar juice is treated with sulphur dioxide, and then caused to flow slowly upwards through a cylindrical tank in which it is heated. By means of a rotating cone near the top of the tank it is forced in the form of a thin whirling film into an annular collecting tank, while milk of lime fed to the upper side of a rotating disc above the cone forms a fine spray which falls on the thin film and is mixed intimately with it. The juice flows successively through other similar tanks in which it is treated with further lime and subsequently with phosphoric acid, the precipitate formed in each tank being separated. By adding the bulk of the lime in the first tank and accurately measured small quantities in the others the juice can readily be brought to exact neutrality. T. S. WHEELER.

Multichamber apparatus for conducting leaching, washing, or like operations on solid or semi-solid material [*e.g.*, removing sugar from plant cuttings]. W. RAABE (U.S.P. 1,575,936, 9.3.26. Appl., 7.2.25).—The material is subjected to countercurrent treatment in a drum provided with partitions to form leaching chambers. Members are provided for lifting the non-liquid material over the partitions, and over surface members, pervious to liquids, arranged adjacent to the partitions. H. HOLMES.

Manufacture of edible sugar from wood. L. PINK (G.P. 425,023, 7.6.22).—Wood pulp mixed with hydrogen peroxide is saturated with chlorine, and, after thorough washing, the product is saccharified by means of dilute acid. L. A. COLES.

Lactose from whey (U.S.P. 1,571,626).—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Comparative effects of pressing and draining yeast. D. H. F. FULLER and F. E. B. MORITZ (J. Inst. Brew., 1926, 32, 171—176).—Drainings or pressings from yeast have a higher content of phosphoric anhydride, nitrogen, and alcohol, than the beer from which the yeast was obtained, the excess representing the amount of these substances lost (or formed) by the yeasts when drained, pressed, or filtered. Pressed yeast loses substantially less nitrogen and phosphorus than does drained or filtered yeast, and less alcohol is found in pressings than in drainings, although the latter are, in view of the temperature, more subject to loss by evaporation. The main factor determining the amount lost by the yeast when it is separated from its surrounding beer is not pressure but the time taken in separation. The temperature, however, also has considerable influence. C. RANKEN.

Industrial manufacture of absolute alcohol. H. GUINOT (Chim. et Ind., 1926, 15, 323—330).—A process is described for the rectification of alcohol based upon that previously recorded (B., 1925, 821). The presence of impurities in the alcohol does not in many cases affect the purity of the final product, and slight modifications are introduced to deal with particular difficulties. In a few instances, notably that of *iso*propyl alcohol, complete separation is impossible by this process. Methyl alcohol as an impurity can be completely removed, and in view of this, the substitution of *iso*propyl for methyl alcohol in denaturants is suggested. The cost of production of absolute alcohol by this means is quite as low, if not lower than that of the ordinary 96% rectified spirit. A. G. POLLARD.

Detection of ethyl phthalate in spirits. R. SCHMITT (Z. Unters. Lebensm., 1926, 51, 56—57).—The test for the detection of ethyl phthalate depending upon the formation of a violet-red colour when it is mixed with pyrogallol and sulphuric acid, is not conclusive. The colour only differs from that produced by pyrogallol and sulphuric acid alone by its redder shade and it is recommended, that, for the detection of ethyl phthalate in spirits and liqueurs, the colour produced by the above test should be compared with that given by a mixture of ethyl phthalate and a pure spirit, adulteration only being proved when the shades of the two colours are identical. E. H. SHARPLES.

Alcohol and other by-products from pea-nut shells. DE BELSUNCE.—See XII.

Action of lime on enzymes. COLLETT.—See XV.

Fermentation of cellulose. ANDERSON.—See XVI.

PATENTS.

Fermenting cellulosic materials. H. LANGWELL (E.P. 248,795, 10.9.24 and 23.10.24).—Improvements in the process described in E.P. 134,265 (B., 1920, 38 A) are claimed. The fermenting mash is so treated as to maintain the hydrogen-ion concentration between the limits of 10^{-9} and 10^{-5}, by the use of suitable phosphates, carbonates, or oxides of alkali or alkaline-earth metals, or of magnesium or ammonium. The cellulosic material may be in the form of naturally occurring products, *e.g.*, fresh grass, bamboo, etc., alone or mixed with sulphite pulp, dry maize cobs, etc. Where green material is used artificial inoculation may be unnecessary. The mass should be stirred or agitated. Dried materials should be pulverised and green material crushed or pulped. A. G. POLLARD.

Process of manufacturing yeast. H. W. DAHLBERG (U.S.P. 1,580,999 and 1,581,000, 13.4.26. Appl., 8.6.22 and 5.3.25).—See E.P. 246,002 ; B., 1926, 295.

Cheese with increased vitamin content (E.P. 226,549).—See XIX.

XIX.—FOODS.

Storage of eggs. T. MORAN and J. PIQUÉ (Food Investigation Special Rep., 1926, [26], 1—61, 62—80 ; cf. B., 1925, 897).—Success in the cold storage of eggs is dependent on the following factors : the

eggs must be fresh and free from cracks either of the shell or of the membrane, these points being determined by "candling"; previous to the storage period the eggs should not be exposed to a temperature exceeding 15°. Cleanliness both of the eggs themselves and of the packing material is essential. The temperature of the store should be maintained at 0—0·6° and the air should be in constant circulation or at least frequently renewed, and the optimum humidity is 80% saturation; a drier atmosphere leads to loss of weight of the eggs by evaporation, a more humid one encourages the growth of moulds. On removal from the store the temperature of the eggs should not be allowed to rise rapidly.

C. R. Harington.

Rapid sorting test for small quantities of tartaric acid in self-raising flour. A. F. Lerrigo (Analyst, 1926, **51**, 180—181).—The flour (about 4 g.) is shaken with 20 c.c. of water, left for 2 min., filtered, and a few drops of dilute ammonia are added, with shaking, to 10 c.c. of the filtrate. About 0·05 g. of powdered silver nitrate is dropped in, and the test-tube placed in water at 70° without shaking. After 2 min. the tube is shaken, when a flour free from tartrate gives a pale yellow turbid liquid, while if tartrate is present the colour produced varies from pale grey to black according to the amount present. Even with 0·1—0·5% of tartrate a mirror is not produced under these conditions.

D. G. Hewer.

Examination of fruit juices. H. Eckart and A. Diem (Z. Unters. Lebensm., 1926, **51**, 48—56).—For the accurate evaluation of fruit juices, in addition to the results of analysis, a series of standards are necessary which must be systematically determined for each harvest period. From the results of numerous investigations of representative samples of raspberry juice, correlating the refractive index, density, sugar content, and acidity, is derived a normal type of raspberry juice (n_D 1·3467, d 1·0374, sugar 9·2% on dry substance, total acid 1·47%) for the 1925 harvest. Abnormal juices and syrups have been examined and the influence of fermentation and dilution with water on the physical and chemical characteristics is fully discussed. The refractometer is recommended for rapid and accurate practical control of fruit juice manufacture. E. H. Sharples.

Determination of pectin. C. F. Ahmann and H. D. Hooker (Ind. Eng. Chem., 1926, **18**, 412—414).—To a solution containing 0·25—1·0 g. of pectin in 200 c.c. a known quantity (50 c.c.) of alkali is added so that the concentration of the alkali will be about 0·1N. The solution is made up to 250 c.c. and kept at 55° for 12 hrs. in a vessel sealed to prevent entrance of carbon dioxide. An aliquot portion is then pipetted off and titrated with hydrochloric acid which should be of about a quarter the strength of the alkali. Taking the neutralisation equivalent of pectic acid at 55° as 194·9 (equivalent to 208·9 g. of pectin) the amount of pectin (x) may be calculated from the amount of alkali used, since NaOH : pectin=weight of alkali combined : x. Results obtained by this method compare well for pure pectin with those obtained by precipitation, but for plant material the titration method is better as it avoids interference of occluded material. The neutralisation equivalent of pectic acid should be determined for the conditions adopted in the determination. D. G. Hewer.

Legumin. M. A. Rakusin and G. Pekarskaja (Z. Unters. Lebensm., 1926, **51**, 43—45).—The preparation and properties of legumin from legumes are described. Its chief reactions compared with those of casein show complete analogy. Like casein, it contains phosphorus, is lævo-rotatory (in pepsin-hydrochloric acid solution), gives the same protein and carbohydrate reactions, is soluble in ammonia and alkalis forming water-soluble leguminates similar in optical properties to the alkali salts of casein and is precipitated from these solutions by dilute acids. The titer, obtained by the determination of silver in silver leguminate, approximates closely to that of casein. E. H. Sharples.

Glass for pharmaceutical purposes. Tirelli. —See VIII.

Determination of milk fat in mixtures. Grossfeld.—See XII.

Determination of coconut oil and milk fat. Baumann, Kuhlmann, and Grossfeld.—See XII.

New value for milk fat. Kuhlmann and Grossfeld.—See XII.

Patents.

Preparation of cheese having an increased vitamin content. Mellemeuropæisk Patent-Financierings-Selskab A./S., Assees. of Hamburger & Co., and H. Liebers (E.P. 226,549, 18.12.24. Conv., 21.12.23).—The vitamin-containing addition consists of yeast which is incorporated in the fresh or liquefied state with the finished cheese. The yeast is pasteurised prior to or after its incorporation so that the proteolytic enzymes are rendered inactive while the other enzymes and vitamins are only slightly detrimentally influenced. Bacteria in the cheese may be destroyed by heat prior to incorporation with the yeast, or proteolytic enzymes of the yeast and bacteria of the cheese may be rendered ineffective simultaneously by kneading the yeast and cheese together under the influence of heat. A cheese of the rindless Emmenthal type is produced by heating the cheese pulp until of a doughy consistence, then cooling to 60—70°, and intimately kneading with fresh or liquefied yeast.

C. Ranken.

Artificial drying of crops. B. J. Owen (E.P. 248,935, 31.3.25. Addn. to 235,273, cf. B., 1925, 648).—The improvement on the prior patent described consists in the arrangement of the supply duct conveying air to the central distributing space within the stack. The duct is bell-mouthed and carried to the distributing space through a channel excavated in the ground beneath the stack.

A. G. Pollard.

Process of obtaining milk sugar [lactose from whey]. H. V. Dunham, Assr. to Rosemary Creamery Co. (U.S.P. 1,571,626, 2.2.26. Appl., 30.1.23).—In the preliminary treatment of whey for the preparation of lactose, the bulk of the albumin is precipitated by boiling the whey at atmospheric pressure by direct fire heat, currents of air being blown against the surface of the liquid to prevent excessive foaming. The boiling is continued until the liquid reaches at least d^{88} 1·16. At this stage the precipitated albumin has been converted into a readily filterable condition and after separating it the whey is further concentrated by boiling at atmospheric pressure.

D. G. Hewer.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Choice of indicators for alkaloidal titrations. H. Wales (Ind. Eng. Chem., 1926, **18**, 390—392).—The range of colour change for a given indicator was found by dissolving 100 mg. of the alkaloid in a slight excess of acid, diluting to 50 c.c., and titrating the excess acid with alkali, using quinhydrone and saturated calomel electrodes and recording voltages at definite intervals. The variation in voltage was plotted against the volume of alkali used, the end-point (centre of break) of the titration determined, and the hydrogen-ion values computed. An indicator is most suitable for a given titration when its mid-point of colour range lies in the region of rapid change in hydrogen-ion value. The following indicators or others of similar range are recommended for the alkaloids named. Morphine, codeine, and nicotine, methyl-red, narcotine, hyoscine, and delcosine, bromophenol-blue, cotarnine, propyl-red or bromocresol-purple, but the yellow colour of the alkaloid may mask the colour of the latter; papaverine is a very weak base and, while no indicator was satisfactory, bromophenol-blue has its colour change at the right point; narceine is apparently completely dissociated in aqueous solution; hydrastine, no indicator satisfactory as the break (in the bromophenol-blue range) is poorly defined; quinine hydrochloride and quinidine, bromocresol-purple and propyl-red; cinchonine, bromocresol-purple to the first change of colour, or propyl-red; strychnine, methyl-red or propyl-red and bromocresol-purple; sparteine, bromothymol-blue for indication of the total amount of acid radical present. D. G. Hewer.

Ether. III. Determination of aldehyde as a contaminant. E. P. Phelps and A. W. Rowe (J. Amer. Chem. Soc., 1926, **48**, 1049—1053; cf. A., 1924, ii, 876).—A standard Schiff's reagent solution is prepared by mixing 30 c.c. of 0·1% aqueous fuchsin solution with 200 c.c. of saturated sulphur dioxide solution, shaking, adding 3 c.c. of concentrated sulphuric acid, keeping the mixture for 24 hrs., and filtering if necessary. Pure acetaldehyde is prepared by depolymerising paraldehyde with dilute sulphuric acid and distilling through a 90-cm. Hempel column. To each of two cylindrical containers 10 c.c. of pure alcohol are added. To one, 1 c.c. of a standard solution of the pure aldehyde in pure ether and 4 c.c. of ether are added, and to the other are added 5 c.c. of the ether sample under examination. The mixtures are kept for 15 min., and if the colours do not nearly match the procedure is repeated using different quantities of the standard aldehyde solution until approximately equal colorations are obtained in the two tubes, which are finally compared in a colorimeter. The depth of colour is not proportional to the concentration of aldehyde present, but by the above matching process accurate determinations can be made, and positive results are given with an aldehyde content of 0·003%. F. G. Willson.

Reagent for the recognition of neosalvarsan unfit for [therapeutical] application. A. Kircher and F. von Ruppert (Pharm. Ztg., 1926, **71**, 264—265). — Strontiuran (strontium chloride–carbamide solution) is described by Hirsch (Mônch. Med. Woch., 1925, 1549) as giving a turbidity with impure (oxidised) neosalvarsan. The appearance or non-appearance of the turbidity, however, depends on the manner in which the neosalvarsan and strontiuran are mixed, different results being obtained according to whether the neosalvarsan is added to the strontiuran solid or in solution, or the strontiuran solution is added to the solid neosalvarsan. Hirsch's further statement that neosalvarsan condemned by his test gives iodine values (c.c. of 0·1*N*-iodine equivalent to 1 g. of neosalvarsan) considerably below 149 is also incorrect, since normal values may be obtained.

B. Fullman.

Acetylation of tannin and analysis of commercial acetyltannins. O. and J. M. Fernández (Anal. Fis. Quím., 1926, **24**, 62—67).—Tannin (pentadigalloylglucose) and digallic anhydride give almost identical products on acetylation. Complete acetylation of tannin, involving the entrance of 25 acetyl groups into the molecule, was effected by the action of excess of acetic anhydride in the presence of small quantities of sulphuric acid. A less completely acetylated product containing 19—20 acetyl groups in the molecule was obtained by acetylating in the presence of a quantity of ethyl acetate equal to the acetic anhydride used. The completely acetylated product is too highly astringent for pharmaceutical purposes. G. W. Robinson.

Examination of commercial tannic acid by comparative methods. W. B. Forbes (Pharm. J., 1926, **116**, 225—229).—Hooper's cinchonine method (B., 1925, 369) and a modification of Crouzel's phenazone method (A., 1903, ii, 113) appear to be the most satisfactory for the determination of gallotannic acid in astringent drugs. The modification of Crouzel's method consists in the addition of the tannic acid to excess of phenazone solution without any sodium bicarbonate; on filtration through a Jena glass filter a clear filtrate is obtained. The precipitate after drying at 100° contains 58% of tannic acid. Four samples of tannic acid B.P. gave by the above methods:—tannic acid 81·7—85·9%, gallic acid 6·4—6·9%, water 8·0—9·38%. Gallic acid was determined by Dreaper's volumetric copper

method (A., 1904, ii, 793), a factor being obtained from experiments with the pure acid, viz., gallic acid = CuO × 0·92. E. H. SHARPLES.

Aldehydes of peppermint oil. R. E. KREMERS (Amer. J. Pharm., 1926, 98, 86—89).—The aldehydes were separated from crude peppermint oil (d 0·869, aldehydes 6%, ester value of aldehyde-free oil 8·2) by means of the bisulphite compounds and fractionated. The fractions had the physical characters of aldehydes of the aliphatic series and the predominating fractions, which were slightly optically active, were within the distilling range of the valeraldehydes. *iso*Valeraldehyde, characterised by conversion into *iso*leucine, was the most important constituent quantitatively. The presence of d-methyl-2-butanal (α-methyl-n-butaldehyde) was not confirmed, and the substance responsible for the optical activity could not be identified. Contrary to the observation of Curtius and Franze (B., 1912, 706), the presence of hexen-2-al-1 ($\alpha\beta$-hexylene-aldehyde) could not be detected. E. H. SHARPLES.

Essential oils from Irish-grown plants. I. Oil of lavender. J. REILLY, P. J. DRUMM, and C. BOYLE (Econ. Proc. Roy. Dublin Soc., 1926, 2, 273—284).—Preliminary investigations on the cultivation of lavender in Ireland. A sample of the mixed oils from *Lavendula vera*, vars. *angustifolia* and *delphinensis* had d 0·8970, ester value 15·8, acid value 0·9, α(100 mm.) —6·3, and n_D 1·4678. These characteristics compare favourably with those of English lavender oil. E. H. SHARPLES.

Determination of ascaridole in chenopodium oil. H. PAGET (Analyst, 1926, 51, 170—176).—In view of the ready conversion by heat of ascaridole into ascaridole glycol anhydride which is easily hydrated (by steam) to ascaridole glycol, these last two substances must be present in chenopodium oil, and since like ascaridole they are miscible with 60% acetic acid, the U.S.P. method of determination of ascaridole must be considered untrustworthy. A reduction method by means of titanous chloride has been worked out whereby 1:4-terpin is formed by addition of 4 atoms of hydrogen to ascaridole. 1 g. of chenopodium oil is diluted to 200 c.c. with 96% alcohol, and to 10 c.c. of this, titanous chloride (66 c.c. of the commercial 15% solution made up to 2250 c.c. and standardised) is added in excess (about 50 c.c.) in a flask through which a current of carbon dioxide is passing. The flask is then closed with a Bunsen valve and the contents are heated almost to boiling point for 1—2 min.; if the pale violet colour disappears more titanous chloride must be added. About 1 c.c. of 5% potassium thiocyanate is then added, the solution titrated with a standard solution of iron alum, and the quantity of titanous chloride oxidised found. Until the products of reduction have been studied 1 g. of ascaridole is taken to be reduced by 1·2770 g. of titanous chloride, which is the mean of several determinations. D. G. HEWER.

Colour reaction of saponin with nitrates. MITCHELL.—See VII.

PATENTS.

Production of unsymmetrical arsenobenzene compounds. DEUTSCHE GOLD- & SILBER-SCHEIDEANSTALT VORM. ROESSLER, and A. ALBERT (E.P. 249,584, 24.11.24).—Readily soluble, unsymmetrical arsenobenzene compounds of therapeutic value, are prepared by reducing, *e.g.*, with sodium hyposulphite, mixtures containing: (i) aromatic aldehydo-arsinic acids, aromatic keto-arsinic acids, or mixed aliphatic-aromatic keto-arsinic acids, or the corresponding arsenoxides, prepared as described in E.P. 199,091 and 220,668 (cf. B., 1923, 862 A; 1924, 966); (ii) condensation products prepared by the action of hydrazine or its derivatives upon the arsinic acids or arsenoxides mentioned above; (iii) condensation products of the arsinic acids or arsenoxides mentioned above, with organic compounds containing reactive amino-groups, other than hydrazine or its derivatives, (cf. E.P. 249,588, following abstract); and (iv) ter- or quinquevalent organic arsenic compounds such as halogen-, nitro-, hydroxy-, or amino-aryl arsenoxides, or carboxy- or sulpho-arsinic acids, or glycine-arsinic acids. The mixtures to be reduced contain approximately equimolecular proportions of two different compounds from groups i, ii, or iii, or of one of the compounds in any of these groups and one compound in group iv. L. A. COLES.

Production of derivatives of organic arseno-compounds. DEUTSCHE GOLD- & SILBER-SCHEIDEANSTALT VORM. ROESSLER, and A. ALBERT (E.P. 249,588, 28.11.24. Addn. to 199,092; cf. B., 1923, 862 A).—The compounds are prepared by the action of organic compounds containing reactive amino-groups, such as hydroxylamine and its derivatives, aniline, carbamide, glycine, p-amino-acetophenone, o-aminobenzaldehyde, and sulphanilic acid, but not including hydrazine and its derivatives, on aromatic aldehydo-arsinic acids or mixed aliphatic-aromatic keto-arsinic acids, or the corresponding arsenoxides. The compounds may subsequently be treated with reducing agents for conversion into the corresponding arsenoxides or arsenobenzene derivatives, without affecting the C:N linkage. L. A. COLES.

Production of alkyl esters [formates]. BADISCHE ANILIN- & SODA-FABR., Assees. of R. WIETZEL (U.S.P. 1,572,698, 9.2.26. Appl., 20.5.25).—A solution of sodium methoxide in methyl alcohol containing about 12% Na is treated at 30—150° with carbon monoxide free from iron carbonyl, sulphur, and water, at pressures of 60—1000 atm. At 80° and 60 atm. 75% of the methyl alcohol is converted into methyl formate in 3 hrs.; at 1000 atm. conversion is complete in a few minutes. The product is withdrawn or distilled from the autoclave, and the sodium methoxide, which is insoluble in methyl formate, remains in the autoclave and is used as catalyst for the next batch. Ethyl formate is prepared in a similar manner. T. S. WHEELER.

Condensation products of compound aliphatic-aromatic ketones with polyalcohols. J. ALTWEGG

and E. F. CHERMETTE, Assrs. to SOC. CHIM. USINES DU RHÔNE (U.S.P. 1,572,176, 9.2.26. Appl., 19.3.24). —Products similar to acetoneglycerol are obtained by allowing alkyl-aryl ketones, *e.g.*, acetophenone, to react with polyhydroxy-compounds in presence of a primary alcohol containing a small quantity of a mineral acid at about 0° for periods of from 1 hr. to 2 days. With excess of ketone practically quantitative yields are obtained. Suitable polyhydroxy-compounds include glycol, glycerin, chlorohydrin, monoacetin, ethyl tartrate and citrate. The products are colourless oils or solids soluble in the usual organic solvents but not in water. They can be distilled under reduced pressure and are of value as solvents, as plasticising agents for cellulose esters, and therapeutically, possessing sedative and slightly hypnotic properties. For example, 450 pts. of ethyl alcohol, 200 pts. of glycerol, and 5 pts. of sulphuric acid are treated at —10° with 500 pts. of acetophenone, and the cold mixture is neutralised with sodium carbonate after 24 hrs. and filtered. The alcohol and excess of ketone are removed by distillation, the latter under reduced pressure. *Acetophenoneglycerol* then distils at 134°/5 mm. It forms a viscous oil d^1 1·159. *Propiophenoneglycerol* has b.p. 140°/5 mm. and d^{15} 1·1255, *acetophenoneglycol* has m.p. 62°, b.p. 130°/40 mm., and *acetophenonemonochlorohydrin* has b.p. 153°/40 mm.

T. S. WHEELER.

Obtaining pyrrole, pyrrole derivatives, and pyrocoll from animal waste. J. MICHELMAN (U.S.P. 1,572,522, 9.2.26. Appl., 11.11.24).—Collagenous tissue such as glue stock from slaughter-houses or leather stock from tanneries is freed from glycerin and glycerides by treatment with lime and heated out of contact with air. The distillate separates into two layers, an aqueous and an oily layer. The oily layer is diluted with toluene or the like, when pyrocoll separates. The liquid is distilled and the fractions to 200° and 200—300° are collected. The first fraction, which contains toluene and pyrrole and substituted pyrroles, is treated with solid potassium hydroxide, when the potassium salts of pyrrole and the substituted pyrroles separate. The higher fraction contains pyrrole derivatives and sometimes hydrocarbon oils, which if present separate as an upper layer.

T. S. WHEELER.

Phenolphthalein. E. B. PUTT (U.S.P. 1,574,934, 2.3.26. Appl., 10.7.25).—100 pts. of pure crystalline phenolphthalein are dissolved in 100 pts. of water, containing half its volume of 50% sodium hydroxide solution, at the ordinary temperature, and the solution is diluted with 1800 pts. of water. Dilute acetic acid and then dilute hydrochloric or other mineral acid are added until the phenolphthalein is completely precipitated, the temperature being maintained below 40°. After keeping for 1 hr. the precipitate is filtered off, washed, and dried at 100° for 8 hrs. Pure phenolphthalein is thus obtained as a white, bulky, amorphous powder, more soluble in alcohol and alkali than crystalline phenolphthalein and possessing on this account greater laxative properties.

T. S. WHEELER.

Preparation of ethyl chloride. CHEM. FABR. VORM. WEILER-TER MEER, Assees. of H. SUIDA (G.P. 420,441, 5.2.24).—Ethyl chloride is prepared by passing a mixture of ethylene, or gases containing it, with hydrogen chloride, under atmospheric or increased pressure, over porous material impregnated with the liquid double compound of aluminium chloride and ethylene, $(C_4H_8,AlCl_3)_x$, obtained by passing ethyl chloride over powdered aluminium chloride at 100—120°, until it is converted completely into a dark brown liquid. On passing a mixture of hydrogen chloride with 95% ethylene through a tube containing the catalytic material at 90°, removing excess hydrogen chloride by washing with water, and recovering the ethyl chloride by cooling and compression, a yield of about 66% is obtained; the yield is higher, however, if increased pressure is used and the temperature may be considerably lower.

L. A. COLES.

Preparation of formaldehyde from methyl alcohol. BADISCHE ANILIN- & SODA-FABR., Assees. of A. BERTRAM and E. KUSS (G.P. 420,442, 29.2.24). —Synthetic methyl alcohol is freed from volatile iron compounds (iron carbonyl) before it is oxidised catalytically to formaldehyde, by passing it, before or after admixture with the air for oxidation, through a tube, 20—80 cm. long, containing inert material, such as glass, quartz, or porcelain, at 150—300°. The iron carbonyl is decomposed, ferric oxide being deposited. The yield of formaldehyde from methyl alcohol vapour containing 19 mg. of iron as iron carbonyl per litre, is increased from 55·8% to 83—85%, and the life of the catalyst is prolonged considerably, by removal of the iron carbonyl.

L. A. COLES.

Production of butyric aldehyde [butaldehyde]. C. E. PIGG, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,576,544, 16.3.26. Appl., 25.7.24).—A mixture of *n*-butyl alcohol vapour with water vapour is passed over a dehydrogenating catalyst at 200—350° and the product is condensed. H. HOLMES.

Preparation of butyric acid. C. W. HANCOCK, Assr. to COMMERCIAL SOLVENTS CORP. (Can. P. 249,868, 30.5.24).—A colloidal solution of manganese butyrate in butaldehyde, spread over material so as to expose a large surface, is treated with a current of oxygen or of gases containing it, until the greater part of the aldehyde is oxidised to butyric acid.

L. A. COLES.

Oxidising aromatic side-chain compounds. P. SEYDEL, Assr. to SEYDEL CHEM. CO. (U.S.P. 1,576,999, 16.3.26. Appl., 16.7.21).—850 pts. of 67% nitric acid, 800 pts. of water, 500 pts. of toluene, and 5 pts. of manganese dioxide are heated to 85° under a pressure of 35—40 lb. per sq. in., and oxygen or a gas rich in oxygen is introduced under pressure at intervals. After 6 hrs. the spent oxides of nitrogen are blown off and fresh oxygen is introduced, this process being repeated at similar intervals until 24

hrs. have elapsed, when the temperature is raised to 110° and the pressure to 75 lb. per sq. in. An 80% yield of benzoic acid is obtained. It contains some nitrobenzoic acids from which it can be separated by sublimation. In place of manganese dioxide, vanadium oxide may be used as a catalyst. The nitrotoluenes can be similarly oxidised.

T. S. Wheeler.

Manufacturing aromatically - disubstituted thioureas [thiocarbamides] of symmetric constitution. W. Flemming (U.S.P. 1,577,797, 23.3.26. Appl., 19.3.25).—An aromatic amine is treated with carbon disulphide and an aqueous solution of a strong base in a closed vessel when the following reaction takes place with evolution of heat :—

$$2RNH_2+2CS_2+2NaOH=SC(NHR)_2+Na_2CS_3+2H_2O.$$

For example, by agitating 100 pts. of aniline, 50 pts. of sodium hydroxide in 150 pts. of water and 82 pts. of carbon disulphide in a closed vessel a yield of over 90% of thiocarbanilide is obtained in 10 min.

T. S. Wheeler.

Preparation of *n*-butyl salicylate. Farbenfabr. vorm. F. Bayer & Co. (F.P. 595,871, 27.3.25. Conv., 11.4.24).—*n*-Butyl salicylate, an oil of b.p. 128°, with a pleasant odour, and of value in the manufacture of perfumes, is obtained by the usual esterification processes. L. A. Coles.

Perfume. Farbenfabr. vorm. F. Bayer & Co. (F.P. 598,002, 12.5.25. Conv., 14.5.24).—The perfume consists of 2:6-dimethylhepten-2-ol-6 or its esters. W. Clark.

Preparation of di- and poly-piperidyls. K. Rülke and F. Clotofski (G.P. 420,445, 20.4.24).—Pyridine and its homologues are reduced electrolytically, with lead cathodes, in 2—3 equivalents of sulphuric acid, of concentration under 30%, preferably 15—25%. For dipiperidyls with little polypiperidyls, the current density should be 8—14 amp./dm.², and for a high total yield of di- and polypiperidyls the current density should be 20 amp./dm.² of cathode surface. For example, technical pyridine bases thus reduced with a current density of 13·6 amp./dm.² at not above 18° yield 32·9% of dipiperidyls, distilling mainly at 155—162°/17 mm. with solidification, and 11·4% of resin. Using a current density of 27 amp./dm.² at not above 30°, the yields are 25·5% of dipiperidyls and 38·7% of resin (polypiperidyls). These products are used for destroying pests. B. Fullman.

Obtaining halogenated hydrocarbons. Badische Anilin- & Soda-Fabr., Assees. of G. Wietzel and J. Dierksen (G.P. 420,500, 29.3.23. Addn. to 409,181).—In modification of the process described in the chief patent (B., 1925, 448), adsorbents which have taken up hydrocarbons are treated with chlorine or bromine. For example, active charcoal in a series of towers (cf. U.S.P. 1,412,954; B., 1922, 454 a) is charged respectively with benzol, sulphur, and ethylene. The sulphur-containing charcoal is treated with chlorine as described in the chief patent (*loc. cit.*). Chlorine is led through the other adsorption-vessels giving respectively chlorinated benzene hydrocarbons, and dichloroethane and its homologues. The latter may, if required, be hydrolysed to, *e.g.*, ethylene glycol, and this converted into ethylene chlorohydrin by means of the sulphur monochloride obtained in the process. The charcoal may be used again after removal of the chlorinated products. B. Fullman.

Preparation of double compounds from aliphatic amino-acids and inorganic alkali salts [phosphates]. C. F. Boehringer & Soehne G.m.b.H. (G.P. 420,910, 24.4.24).—Double compounds of, *e.g.*, glycine, leucine, or alanine with disodium phosphate, or of glycine with dipotassium phosphate or trisodium phosphate, are prepared by combining stoichiometric proportions of the constituents in aqueous solution, with subsequent isolation in the solid form by evaporation, salting out, or precipitation with alcohol. Solutions of the compounds, after sterilisation, are suitable for injection in cases of fractured bones, being better than sodium phosphate for this purpose, as they do not cause rise in temperature, headache, or sickness. L. A. Coles.

Preparation of pure α-sparteine methiodide. E. Merck, Chem. Fabr., Assees. of A. Dützmann (G.P. 421,387, 23.8.24).—The methiodide prepared from the crude sparteine base is dissolved in twice its volume of alcohol, and treated warm with an equal volume of ethyl acetate. After keeping for 1 day the crystalline product is filtered off, and freed from chloroform by solution in water and boiling. On cooling pure α-sparteine methiodide crystallises out (compound with ½ $CHCl_3$, m.p. 236—238°). The mother liquor yields α'-sparteine methiodide.

B. Fullman.

Preparation of a complex thorium compound of the reaction product of tyrosine and formaldehyde. Chem. Fabr. Flora (Swiss P. 113,053, 13.11.24).—The product obtained from the reaction between tyrosine and 40% formaldehyde in the presence of 30% sodium hydroxide is treated in aqueous solution with acetic acid and excess of freshly precipitated thorium hydroxide. The solution, after warming, filtering, and concentrating, is treated with ether, whereby a yellow amorphous complex compound, containing 10% Th, is precipitated. It has therapeutic application.

B. Fullman.

Pharmaceutical product. J. Callsen, Assr. to Winthrop Chem. Co., Inc. (U.S.P. 1,572,768, 9.2.26. Appl., 26.6.25).—Aminoalcohols are treated with chloroquinoline derivatives in presence of sodium to yield basic ethers of quinoline which form mono- and di-hydrochlorides and are of therapeutic value in the treatment of diseases of the heart. For example, 178 pts. of dimethylaminoethyl alcohol are dissolved in 800 pts. of xylene, 46 pts. of sodium and 355 pts. of α-chloro-γ-methylquinoline are added, the mixture is heated to 100°, filtered from sodium

chloride, and the filtrate treated with dilute hydrochloric acid. The precipitated hydrochloride is treated with alkali and the free base distilled under reduced pressure. *α-Hydroxy-γ-methylquinoline dimethylaminoethyl ether* thus obtained has b.p. 176°/10 mm., is soluble in the usual organic solvents and insoluble in water; the *dihydrochloride* has m.p. 163° and the *monohydrochloride* m.p. 190°. *α-Hydroxy-γ-methylquinoline piperidoethyl ether* from α-chloro-γ-methylquinoline and piperidoethyl alcohol has b.p. 233°/17 mm., *dihydrochloride* m.p. 187°. *γ-Hydroxy-α-methylquinoline dimethylaminoethyl ether* from γ-chloro-α-methylquinoline and dimethylaminoethyl alcohol has b.p. 190°/10 mm.

T. S. WHEELER.

Organic derivatives of tin. C. OECHSLIN, Assr. to ETABL. POULENC FRÈRES (U.S.P. 1,573,738, 16.2.26. Appl., 4.9.24).—225 pts. of stannous chloride dissolved in 380 pts. of water are mixed with about 700 pts. of sodium hydroxide solution, *d* 1·36, and 300 pts. of ice, and treated with 80·5 pts. of ethylene chlorohydrin. The sodium salt of hydroxyethyl stannonic acid $OH·CH_2·CH_2·SnO_2Na$ is formed. It is unstable and is preferably isolated as its thio-derivative, by adding barium chloride and saturating with hydrogen sulphide, neutralising, keeping overnight, filtering, and adding acetic acid. The hydroxyethylthiostannonic acid $HO·CH_2·CH_2·SnS·S·SnS·CH_2·CH_2OH$, so obtained, decomposes on heating to 80° yielding tin sulphide and oxysulphide, or on treatment with acids. Glycerol monobromohydrin gives dihydroxypropylstannonic acid which is also isolated as the thio-derivative. T. S. WHEELER.

Polymerisation of butaldehyde. C. BOGIN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,576,503, 16.3.23. Appl., 4.6.24).—*n*-Butaldehyde treated with 0·5—2% of a mineral acid or zinc chloride or the like at 15—50° gives an 85—90% yield of parabutaldehyde, b.p. 210—220° (decomp.) at ordinary pressure, 129—130°/35 mm., d^{21} 0·917. Zinc chloride is particularly suitable as condensing agent as no cooling is then required. The condensation is complete in 4 days and after washing out the catalyst the product is distilled under reduced pressure. T. S. WHEELER.

Preparation of a derivative of 1-phenyl-2 : 3-dimethyl-5-pyrazolonyliminopyrine. KNOLL & Co., and H. BOIE (G.P. 420,446, 28.12.23).—The compound is obtained by fusing together, or by bringing together in solution, equimolecular proportions of the iminopyrine and salicylic acid. For example, the constituents are heated with alcohol until dissolved, and, after cooling, the salicylate is precipitated as crystals containing 3 mols. of water, m.p. 70—71°, by the addition of ether. The compound is only slightly soluble in water, but by the addition of carbamide, urethane, sodium salicylate, or glycerin, concentrated solutions can be prepared suitable, *e.g.*, for intravenous injection in treating *arthritis deformans*. L. A. COLES.

Preparation of ethers of aliphatic or aromatic dialkylaminoethanol esters and their derivatives. W. SCHOELLER (G.P. 420,447, 20.4.23).—Allyl esters of aliphatic or aromatic acids are treated with mercury salts of oxygen acids in alcoholic media, the resulting complex mercury compounds being converted, preferably in non-aqueous media, via their halogenated mercury derivatives into the corresponding halogen derivatives, and these, by the action of secondary aliphatic amines, into dialkylaminoethanols or their derivatives. For example, allyl acetate on treatment with mercury acetate in methyl alcoholic solution yields the corresponding *mercuriacetate*, which on treatment with potassium bromide yields *α-aceto-β-methoxypropane γ-mercuribromide* (the corresponding *β-ethoxy*-derivative being obtained in ethyl alcoholic solution). Action of iodine gives *α-aceto-β-methoxy-γ-iodopropane*, b.p. 82—83°/1·5 mm., leading to *α-aceto-β-methoxy-γ-diethylaminopropane*, b.p. 99—102°/14 mm. The corresponding dimethylamino-compound forms a *methiodide*, m.p. 157°. The following were also prepared :—*α-benzoyl-β-methoxypropane γ-mercuri-iodide*, the corresponding γ-iodopropane *derivative*, b.p. 95—98° *in vacuo*, and the *diethylamino-derivative*, b.p. 100° *in vacuo*; *α-p-nitrobenzoyl-β-methoxypropane γ-mercuribromide*, m.p. 100—102°, the corresponding syrupy iodopropane *derivative*, and the *hydrochloride* of the *diethylamino-derivative*, m.p. 143—144°, reduction of the latter yielding *α-p-aminobenzoyl-β-methoxy-γ-diethylaminopropane hydrochloride*, m.p. 158—159°. The ethers of diaminoethanol esters are effective in local anæstheisa and in increasing intestinal peristalsis.

B. FULLMAN.

Removing nicotine from tobacco. T. SCHLOESING (E.P. 234,845, 29.5.25. Conv., 30.5.24). —Tobacco is freed from nicotine by subjecting it at about 100° to the action of a current of a mixture of steam and ammonia, in which the ammonia is maintained at a constant pressure. The vapour leaving the apparatus is condensed, nicotine is extracted from the condensed liquid by treating it with a nicotine solvent, *e.g.*, kerosene, and the residual liquor, which contains ammonia and small quantities of the aromatic constituents of the tobacco, is boiled, and the steam generated is returned to the process, further quantities of ammonia being added, if necessary. Nicotine is recovered from the kerosene solution, *e.g.*, by extraction with sulphuric acid, and the kerosene is used again. L. A. COLES.

Tobacco denicotinisation. H. W. SMITH (U.S.P. 1,577,768, 23.3.26. Appl., 7.6.22).—Tobacco is treated first with steam at 100—150° and a pressure of up to 55 lb. per sq. in., and then with a volatile base such as ammonia or methylamine. The nicotine thus liberated is extracted with an organic solvent, from which it is recovered by treatment with aqueous acid. The solvent freed from nicotine but saturated with other substances extracted from the tobacco is used for treating succeeding batches. The denicotinised tobacco is treated with steam to remove the volatile base and traces of the solvent. The nicotine content of tobacco can in this way be reduced to 0·4—0·2%. T. S. WHEELER.

Production of acridinium compounds. L. CASSELLA & Co., Assees. of L. BENDA and O. SIEVERS (U.S.P. 1,565,891, 15.12.25, and 1,577,415, 16.3.26. Appl., 22.12.23 and 18.4.25).—See E.P. 217,715 and 218,542 ; B., 1924, 768, 808.

Manufacture of acetic anhydride and aldehyde. G. J. ROY, Assr. to Soc. CHIM. USINES DU RHÔNE (U.S.P. 1,579,248, 6.4.26. Appl., 10.7.25).—See E.P. 238,825 ; B., 1925, 900.

Heart-affecting pure glucoside from ***Bulbus scillæ.*** CHEM. WORKS FORMERLY SANDOZ, Assees. of E. SUTER (U.S.P. 1,579,338, 6.4.26. Appl., 26.5.24).—See E.P. 217,247 ; B., 1924, 997.

Producing a preparation for treating arteriosclerosis. V. MLADEJOVSKY (U.S.P. 1,579,828, 6.4.26. Appl., 12.12.24).—See E.P. 237,087 ; B., 1925, 784.

Manufacture of 1-phenyl-2:3-dimethyl-4-dimethylamino-5-pyrazolone. G. LOCKEMANN (U.S.P. 1,580,059, 6.4.26. Appl., 31.3.24).—See E.P. 214,261 ; B., 1925, 378.

Injectable, homogeneous, oily preparation of bismuth [naphthenate]. W. EICHHOLZ and O. DALMER (U.S.P. 1,580,592, 13.4.26. Appl., 26.11.24). See E.P. 226,226 ; B., 1925, 737.

Oxidation of organic substances (G.P. 420,444). —See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitisers of gelatin. A. AND L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1925, iii., **12**, 338—341).—In 1906 the authors removed by repeated extraction with cold water the sensitising properties from a gelatin which gave very fast emulsions. The residues remaining after evaporation of the extracts were very active, and sensitised inactive gelatins, especially those which had been rendered insensitive by washing. The residue contained inorganic matter and organic substances containing sulphur and nitrogen. The nitrogen content of the initial gelatin decreased on washing. In 1910, 900 substances were examined for their effect on the sensitivity of *X*-ray emulsions. Among those which were selected as having practical value in increasing or decreasing sensitivity or modifying gradation were : æsculine, salts of codeine and thebaine, diethylenediamine, thiocarbamide, guanidine thiocyanate, salts of vanadic acid, and salts of copper. W. CLARK.

Silver iodide in photographic emulsions. W. D. BALDSIEFEN, V. B. SEASE, and F. F. RENWICK (Phot. J., 1926, **66**, 163—184).—A formula is given for the preparation of a silver iodide emulsion having a wide range of grain-sizes and free from clumps. The emulsion was separated by sedimentation into a number of fairly uniform emulsions of different grain sizes, and the effect of its addition to silver bromide emulsions at various stages of manufacture was studied. It is established that silver iodide emulsion can be substituted, before mixing, for the equivalent amount of potassium iodide in an iodobromide emulsion without producing any essential change in photographic properties, turbidity, or distribution of silver iodide in the bromide. This holds for both double neutral emulsification and single ammonia emulsification processes, and for 2% and 3·75% of iodide. The size of particle of the silver iodide emulsion used is without influence, the quantity only being important. Emulsions containing 2% of iodide are slower and more "contrasty" than those containing 3·75%. If silver iodide emulsion is added immediately after the first emulsification in the case of a double emulsification emulsion or after mixing in the case of a single emulsification emulsion or a single ammonia emulsification emulsion, slow emulsions are obtained, and the iodide is fairly uniformly distributed in all layers of the sedimented emulsion. It is concluded that when silver iodide is added to a freshly precipitated silver bromide emulsion the latter is able to peptise the silver iodide grains and form mixed iodobromide grains with them. A fully ripened silver bromide is unable to peptise silver iodide. The silver iodide added just after emulsification exerts a true desensitising effect rather than an anti-ripening influence. The substitution of silver iodide prepared with a slight excess of silver ions for that prepared with excess iodide ions gave the same results. As the time of addition of silver iodide is delayed in the process of manufacture of a double emulsification emulsion, there is a progressive speed decrease for constant time of ripening. The most sensitive emulsion is obtained if the iodide is added just before the first mixing; the lowest speed if the iodide addition is made just after the second mixing. The desensitising action of potassium iodide added after mixing to both single and double emulsification emulsions is no greater than that of silver iodide. The size of grain of the silver iodide emulsion used has no influence on the photographic characteristics of the resulting silver iodobromide emulsion. If coarse, dried silver iodide prepared in absence of gelatin is used, the resulting emulsions are much less sensitive than those made with potassium iodide. If freshly precipitated, wet silver iodide made in absence of gelatin is employed, results similar to those found with silver iodide emulsion or potassium iodide are obtained. The presence of gelatin in the silver iodide is not necessary for it to behave like potassium iodide, although the physical condition is of vital importance. Ripening is assumed to be accompanied by a change of distribution tending to bury the iodide within the bromide. W. CLARK.

Characteristics and anomalies of emulsions on development. I. S. E. SHEPPARD (Phot. J., 1926, **66**, 190—200).—The paper is mainly a plea for a definite classification of photographic emulsions of reproducible type, capable of specification, and for the elimination as far as possible of emulsions not meeting the specifications. The variability of

behaviour of emulsions is considered, and data in this connexion are given. Emulsions are classified, according to their behaviour on development for a series of different times, as orthophotic and anorthophotic. Orthophotic emulsions are those in which the straight-line portions of the family of characteristic curves meet at a definite single convergence point either below, upon, or above the log E axis. Variations of emulsions within the limits of orthophotic types are discussed, and it is shown that all industrially employed emulsions, including those of portrait type, can be produced within this restriction. Anomalous kinds of emulsions have a lower degree of complete reproducibility, *i.e.*, of giving an identical family of characteristic curves for different times of development. These emulsions are less amenable to application of the laws of tone reproduction, to automatic processing, and to application in science and technology. Anorthophotic emulsions are not necessarily consequences of the complexity of emulsion making. W. CLARK.

Supposed connexion between sensitisation of silver halide emulsions and the bleaching of dyes. S. E. SHEPPARD (Phot. Ind., 1926, 301).—There is no connexion between the sensitisation of the bleaching of dyes by thiocarbamides and the sensitisation of photographic emulsions by such compounds. In the latter case sensitising occurs only after decomposition of the thiocarbamide-silver bromide complex. Salts and esters of thiocarbonic acid and dithiocarbonic acid are all photographic sensitisers, but have little or no action on the bleaching of dyes. Numerous criticisms of the silver sulphide theory of sensitivity are dealt with. W. CLARK.

Chemical [photographic] sensitisers. G. KÖGEL and A. STEIGMANN (Phot. Ind., 1926, 355).—Further evidence dealing with the hydrogenation theory of sensitisation is given. W. CLARK.

Sensitivity of photographic papers.—MAUGE (Bull. Soc. Franç. Phot., 1925, iii., **12**, 366—381).—Data are given of the relative speeds of 27 commercial papers, measured by the threshold value, using 5 different metol-quinol development formulæ. The speed of any one paper varies in an irregular manner, according to the composition of the developer, some papers showing considerable variation and others practically none. Using a developer of composition based on the mean proportions of the five formulæ, speeds of practically the mean values were obtained. Variations permissible in a formula without appreciably affecting the results are discussed. Except for special baths, sodium carbonate should be the only alkali used in a typical developer. Addition of desensitiser to prevent aerial fog is advised. The paper speeds are also given for a diaminophenol developer, and lie in general above those found with metol-quinol. Results with iconyl (p-hydroxyphenylglycine) agree practically with those obtained with metol-quinol. Actual and "useful" sensitivities are discussed. For a constant light source and constant development, a variation of exposure time of $\pm$ 15% is permissible for obtaining a correct print. Variations of sensitivity among papers of one type are of most importance in the case of "contrasty" papers. Speed and contrast characteristics of 31 French papers are tabulated. W. CLARK.

Arrest of development in [photographic] plate testing. S. O. RAWLING (Phot. J., 1926, **66**, 187—189).—Neither acid fixing baths (thiosulphate, sodium sulphite, acetic acid, potash alum) nor acetic acid-alum baths exert any noticeable solvent action on the developed (pyrogallol-soda) image on a photographic plate. Comparative tests of the two baths and a water spray for their efficiency in arresting development showed that the two acid baths are equally efficient, while the water spray permits considerable development to go on after removal of the plate from the developer. After long times of development the differences between the methods are negligible. W. CLARK.

Photographic photometry with intermittent exposure from the standpoint of a new blackening law. H. M. KELLNER (Z. wiss. Phot., 1926, **24**, 41—62).—A formula for the blackening of a plate when a rotating sector is used is evolved and tested. The Schwarzschild exponent, p, varies with i, t according to the relation $p = a + b\,e^{-c[\log i t]^2}$, where a, b, and c are constants. An explanation of the decrease of p to a constant minimum with increasing $i\,t$ is advanced. The following "general blackening law" is deduced: $S = m\,e^{-g\sqrt[3]{it^p}}$ where g and m are constants, and determine the contrast and maximum density respectively. Latent image formation is ascribed to liberation of electrons from bromine ions having deformed electron orbits in the silver bromide lattice, the degree of "excitation" being distributed among the bromine ions according to Maxwell's law. Ripening consists in the formation of deformed electron shells. In optical sensitisation the sensitiser is probably arranged on the grain surfaces at the points occupied by the highly-excited bromine ions, and on exposure the dye complex and not the bromine yields electrons to the silver ions. A knowledge of the variation of p with $i\,t$ is necessary for accurate photographic photometry, and ignorance of this invalidates much earlier work. W. CLARK.

Small mercury vapour lamp. CLARK.—See XI.

PATENT.

Preparation of stripping films. A. VEITINGER (G.P. 422,913, 25.12.23).—Gelatin or similar colloid films are provided with a substratum of gelatin or other similar substance prepared by treating the gelatin etc. with oxidising agents, metal salts (with the exception of light-sensitive chromium salts), hardening or tanning agents, or dehydrating agents, or with ultra-violet or other chemically active rays, with subsequent exposure, washing, and drying. The gelatin under-film can be prepared without the use of glass, *e.g.*, on a paper base. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Apparent viscosity of solutions of nitrocotton. McBain, Harvey, and Smith.—See V.

PATENTS.

Removing colouring matter from smokeless powder. E. C. Pitman and G. F. Hunter, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,577,075, 16.3.26. Appl., 20.11.22).—Colouring matter is removed from "pyro" smokeless powder by decomposing the insoluble colouring matter by a dilute acid with formation of soluble products, which are then extracted by a solvent.

S. Binning.

Manufacture of detonating compositions. H. Rathsburg (U.S.P. 1,580,572, 13.4.26. Appl., 3.8.22).—See E.P. 195,344; B., 1923, 804 a.

XXIII.—SANITATION; WATER PURIFICATION.

Purification of water and action of various waters on lead and copper pipes. J. C. Thresh and J. F. Beale (Water and Water Eng., 1925, **27**, 475—478).—Data derived from operation of a 25,000-gall. experimental plant for treatment of water from the Rivers Chelmer and Blackwater are given. The purification process consisted of excess lime treatment, recarbonatation, and rapid sand filtration. Sufficient lime was added to maintain an excess of 1 pt. per 100,000 after 24 hrs., the process being controlled by reaction to brilliant-cresyl-blue. With this excess the total number of bacteria was reduced from thousands to units, *B. coli* was invariably absent in 100 c.c., and organic matter was reduced 50%. To obtain rapid precipitation and decolorisation it was necessary to add, prior to the lime, 1 grain per gall. of aluminium sulphate. Lime water was more efficient and economical than milk of lime. Laboratory experiments have shown that 1 pt. of phenol in 5000 million parts of water will give rise to taste. Potassium permanganate is ineffective for destroying this taste, but an extremely small quantity of ammonia added with the chlorine appears to prevent its formation. *B. coli* was found to flourish in the presence of the weed *Enteromorpha intestinalis*, which is usually found in brackish water but which in this case was growing abundantly in the reservoir and filter beds. Similar multiplication occurred in presence of *Oscillatoria nigra*. Decaying weeds give rise also to an enormous increase of *B. aerogenes*. The presence of *B. coli* is thus not always evidence of manurial pollution. The metals zinc, iron, lead, and copper are slowly oxidised by dissolved oxygen in water. Lead oxide is fairly soluble, and as the action of lead on the human system is cumulative, its presence in drinking water even in minute amounts is dangerous. Copper oxide is much less soluble and there is no evidence of cumulative effect. The action of water on lead is influenced by the saline content of the water, but this does not appear to be a factor in the case of copper. Addition of sodium silicate to water prevents the oxidation of lead, and addition of a trace of carbonate converts any oxide formed into oxycarbonate, which coats the metal and prevents further action. Metal used for tinning copper pipe for use in acid waters should contain 99% of tin; pipe lined with 50 : 50 lead–tin alloy is attacked by acid water nearly as vigorously as lead alone.

R. E. Thompson.

Biology of Jersey water works. W. Rushton and P. A. Aubin (Water and Water Eng., 1925, **27**, 487—488; cf. B., 1925, 260).—Microscopical examination of the water showed that the dominant diatom present was *Asterionella*. Contrary to reports of most observers that this organism in numbers exceeding 2000 per c.c. gives rise to a geranium or fishy odour, numbers as high as 31,184 per c.c. were recorded without perceptible taste or odour. It is suggested that absence of taste and odour may be due to the high chloride content of the water, which is normally 7·5 pts. per 100,000. During one period the number of *Asterionella* present interfered with filtration and it was necessary to apply copper sulphate. Continued employment of the spraying method of applying copper sulphate has demonstrated its efficacy over other methods. Temperature does not appear to be the determining factor in the increase of diatoms, which frequently follows an increase in inflow of soil water. R. E. Thompson.

[Coagulation in water purification.] A. V. De la Porte (43rd Annual Rept., 1924, Provincial Bd. of Health, Ontario, 1925, 112—114).—Laboratory experiments on the use of sodium aluminate as a coagulant were fairly successful with highly acid waters, but few waters were found which were sufficiently acid to precipitate completely the amount required for efficient filtration. When solutions of alum and soda ash of certain strengths were added together, the first precipitate of aluminium hydroxide dissolved, and when the resulting solution was added to water an aluminium hydroxide floc was obtained immediately. Laboratory experiments on coagulation of a number of different types of water by this method have given excellent results.

R. E. Thompson.

Zeolite water softening. F. B. Beech (Eng. and Cont., Water Works Issue, 1925, 64, 1051—1054).—Experiences with a zeolite water-softening plant for treating boiler feed water are outlined. The saving in fuel consumption and boiler maintenance effected during the first 10 months of working was sufficient to pay the cost of the softening plant. The base-exchange compound used is glauconite (greensand), a hydrous silicate of iron, aluminium, and potassium, and after 20 months' use the only important change detectable in the material was an increase in the manganese content from 0 to 0·28%. Approximately 1/3 lb. of sodium chloride is required for regeneration per 1000 grains of hardness removed. The plant will remove manganese and *Crenothrix*, rendering the water suitable for laundry purposes etc. There is little difference in the cost of softening by this method and by the lime-soda process.

R. E. Thompson.

Chlorine as a sterilising agent for water, especially for flood-water. R. Schwarzbach (Gas- u. Wasserf., 1926, 69, 272—275).—Sterilisation is especially necessary in the case of surface water supplies in times of flood when the operation of filter-beds may be deranged. The use of bleaching powder has several disadvantages which led to a chlorine installation being substituted for it. In this apparatus chlorine derived from a cylinder is dissolved in water and this water added to the main water supply in the proportion desired. Bacterial counts are given showing a superior result for chlorine as against bleaching powder, attributed to the greater facility of control of the former. C. Irwin.

Significance of bacteriophage in surface water. L. Arnold (Amer. J. Pub. Health, 1925, 15, 950—952).—Bacteriophagic substances in variable amounts were found in the water of the Chicago River, the concentration being in direct proportion to the amount of pollution by domestic sewage. The bacteriological examination of water may be influenced by the content of bacteriophagic substances in the water. Impregnation of water filters with bacteriophagic substances active against certain pathogenic bacteria offers a new field of investigation in water purification. R. E. Thompson.

Hydrogen-ion concentration—its meaning and its application [in water purification and water bacteriology]. W. A. Taylor (Eng. and Cont., Water Works Issue, 1926, 65, 15—20).

Electrical conductivity and hydrogen-ion control of waste [sewage] disposal. H. C. Parker (Water and Water Eng., 1925, 27, 445—448).—Recent improvements and developments in apparatus for determining p_H (cf. B., 1925, 555, 696) are discussed, and their practical application in the control of the direct oxidation process of sewage treatment is described. R. E. Thompson.

Treatment of sewage at Manchester. E. Ardern (Surveyor, 1925, 68, 465).—Data on the operation of activated sludge units and contact beds at Manchester are given. Dewatering of sludge produced in the treatment of tank effluent by the activated sludge process yielded disappointing results, but preliminary treatment with alum of sludge derived from the treatment of raw screened and detritus-free sewage by this method was more encouraging. Sufficient alum was added to allow of using the maximum rate of filtration, as determined by laboratory tests with a Büchner funnel. Quantities of alum in excess of this amount produced a drier product but did not materially alter the rate of filtration. Adjustment of p_H by acid alone yielded unsatisfactory results, but economy was effected by use of acid with the alum when the sludge leaving the aeration tanks was in good condition. Trade wastes continued to cause difficulty, the greatest disturbance being caused by presence of oily and tarry matter, which retards the activity of the sludge and results in the production of turbid effluents. R. E. Thompson.

Activated [sewage] sludge experiments at Bradford. J. A. Reddie (Surveyor, 1925, 68, 451—453).—Tests carried out during two 30-day periods in an experimental activated sludge tank treating Bradford sewage, which has an abnormally high nitrogen content, to determine nitrogen losses, gave the following results: as free and saline ammonia, 71·3 and 100% loss; as albuminoid ammonia, 2·07 and 14·1% gain; as nitrate, 100 and 100% loss; in other forms 34·3 and 34·2% gain. The total nitrogen loss was 8·6 and 18·6%, respectively. Experiments were also carried out to determine the value of the activated sludge system as a partial process, either as final treatment after preliminary tank treatment, or as a preliminary to a high-rate filtration process. It was found that the effluent from an experimental activated sludge tank could be passed on to filters at 4 times the rate that ordinary tank effluent can be satisfactorily treated, whereas tank effluent of similar strength, produced by secondary precipitation, could not be successfully treated at even double the rate. During one period of the operation of the experimental unit, analyses of the influent and effluent indicated a reduction of 91·2% of the solids entering the tank and almost complete destruction of grease, a constituent which amounts to 40—50% of the dry solids of the Bradford sewage. This destruction is attributed to the presence in the sewage, during the period, of a very large proportion of liquefying bacteria, which varied from 30 to 80% of the total count, compared with 15—20% under normal conditions. R. E. Thompson.

Heat-drying of [sewage] sludge. C. E. Keefer (Eng. News-Rec., 1926, 96, 238—240).—A review of the experience during 6½ years at Baltimore, U.S.A., of heat-drying (rotary dryer) and marketing of sludge as a means of disposal. The results obtained indicate the method to be an expensive one to use under the conditions now existing at Baltimore, and when dealing with sludge of low nitrogen content. The material treated was air-dried sludge containing 68% of water. The product contained 10—20% of water and approx. 2% of nitrogen as available ammonia. If the sludge had contained about 4% of nitrogen as available ammonia, the plant could possibly have been operated at a slight profit. Agglomeration of the sludge into balls was guarded against by using care in feeding, and the warping of the end of the dryer nearest the furnace was prevented by increasing the draught of air. W. T. Lockett.

Sewage tank gases. A. M. Buswell and S. I. Strickhouser (Ind. Eng. Chem., 1926, 18, 407—409).—Gases evolved from foaming Imhoff tanks were found to differ from those from non-foaming tanks in having a higher content of carbon dioxide and a lower content of nitrogen. The presence or absence of hydrogen in gases from septic tanks may be influenced by the proportion of cellulose in the tank, the type of anaerobic decomposition occurring and the using or giving up of any hydrogen so formed, and bacterial formation of methane and water from

hydrogen and carbon dioxide. The gas evolved from Imhoff tanks was found to have sufficient heating power (about 700 B.Th.U. per cub. ft.) to warrant its collection for fuel purposes. D. G. HEWER.

Report on sewage sludge. L. PEARSE and others (Amer. J. Pub. Health, 1926, **16**, 39—42).—The report deals mainly with the utilisation of activated sludge. As a temporary measure activated sludge is being digested at Indianapolis in tanks with primary sludge produced by fine screening and rapid settling. Digestion of activated sludge with primary sludge in Imhoff tanks is being carried out in the Ruhr district, Germany, and in the Sanitary District of Chicago. Imhoff proposes to dispose of sludge in primary digestion tanks and to utilise the gases formed as power for compressing air required for the purification process. As reagents for the conditioning of sludge for filtration or pressing, aluminium sulphate is used at Chicago, sulphur by Houston, and sulphuric acid at Milwaukee. Recent experiments at Chicago indicate that ferric chloride is more efficient but more expensive. Artificial heating of sludge prior to de-watering has not been found necessary at Chicago. R. E. THOMPSON.

Determination of the activity of commercial disinfectants. H. LÜERS and F. WEINFURTNER (Woch. Brau., 1926, **43**, 25—29, 35—39, 45—49).—A modification of the Rideal-Walker test was used, with brewers' yeast as substrate. The figures given below represent the reciprocals of the percentage concentrations at which the various disinfectants had a toxic action on yeast suspensions equal to that of a 1% solution of phenol. Ammonium bifluoride 0·4, hydrofluosilicic acid 0·4, "Pyricit" (chiefly sodium borofluoride with acid sodium sulphate) 0·6, formaldehyde (36%) 0·9, formic acid 1·6, "Antiformin" and "Radoform" (solutions containing 5—7% each of sodium hydroxide and hypochlorite) 33, "Mianin" (sodium *p*-toluenesulphochloramide with 10% active Cl) 5, benzoic acid 5, "Activin" (similar to "Mianin") 8, "Chloramin" (similar to "Mianin" but purer, with 12·6% active Cl), 9, "Pantosept" (sodium salt of *N*-dichloro-*p*) sulphamidobenzoic acid, with 16·8% active Cl-11—13, "Novocit" 12—13, sulphurous acid 25, "Magnocid" (basic magnesium hypochlorite, with 32% active Cl) 30, salicylic acid 35, bleaching powder 35, active chlorine 65, *cyclo*tellurodimethylpentan-3 : 5-dione (impure) 80, mercuric chloride 110, "Caporit" (crystalline calcium hypochlorite with 47% active Cl) 120. J. H. LANE.

Bactericidal properties of the quinone group. HILPERT.—See A., Mar., 321.

PATENTS.

Sewage purification plant. M. PRÜSS (E.P. 247,066, 13.6.25. Addn. to 235,872; cf. B., 1926, 302).—A settling basin and a digestion chamber comprising a sewage purification plant, are hydraulically separated by a thin wall of heat-permeable material, the walls of the digestion chamber, which extend upwards, being well insulated. The settling basin is divided by partitions into three compartments, and in order to prevent floating up of the sewage, the liquid level in the middle part is kept at a different height from that in the outer parts by a system of gutters and off-flows. The sludge is raised from the bottom of the settling chamber to the digestion chamber by an air lift operated by a supply of compressed air, and serves to keep the contents of the digestion chamber agitated by flowing along guiding walls fixed in the chamber. B. W. CLARKE.

Method of killing micro-organisms. W. RUPPEL, Assr. to AMER. ELECTRO-OSMOSIS CORP. (U.S.P. 1,577,659, 23.3.26. Appl., 4.8.21).—See E.P. 150,318; B., 1921, 673 A.

Base-exchanging materials for softening water, from clay. PERMUTIT A.-G. (G.P. 423,224, 18.11.16).—Clay in the form of granules of 2—4 mm. diameter, is ignited at 500—550°. E. S. KREIS.

Sterilising water and other liquids. SIEMENS & HALSKE A.-G., Assees. of G. ERLWEIN (G.P. 422,902, 10.5.24).—Liquefied ozone is used. The liquid ozone may be led direct from the ozone condenser to the mixing chamber, where it is warmed by the water and converted into gaseous ozone, which dissolves more readily and is more active than ozonised air. E. S. KREIS.

Apparatus for the purification of sewage with activated sludge. K. IMHOFF (G.P. 418,319, 21.8.23).—The floors of an aerating chamber and of a settling chamber in the upper part of it, are sharply inclined and so constructed that the solid material from both chambers settles continuously into the lowest part of the aerating chamber, where there are devices for aerating the sewage and circulating the sludge. L. A. COLES.

Further decomposition of partially decomposed sewage sludge. H. BACH (G.P. 421,160, 10.2.24).—Further decomposition of sewage sludge, in which decomposition of the organic material has practically ceased, is aided by addition to the sludge of material containing sugars, such as dairy waste or liquor from beet-slicing machines. L. A. COLES.

Clarification and decolorisation of waste water from dyeworks. G. ULLMANN (Austr. P. 100,735, 10.10.24).—The water is subjected to vigorous atmospheric oxidation, dyes are precipitated by the addition of alkalis or by salting out, and, after filtration through a rapid filter, the last traces of dyes are absorbed from the water by treatment with finely-divided material such as wood or straw. L. A. COLES.

Base exchange materials. H. J. C. FORRESTER. From INTERNAT. FILTER CO. (E.P. 248,070, 26.11.24).—See U.S.P. 1,515,007; B., 1925, 24.

Methods of, and means for, exterminating vermin. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER UND CO. (E.P. 247,249, 10.10.24).—See G.P. 387,988; B., 1924, 804.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 25, 1926.

I.—GENERAL; PLANT; MACHINERY.

Modified fusion pot. V. S. Babasinian and R. D. Billinger (Ind. Eng. Chem., 1926, 18, 340).—A nickel vessel for the fusion of organic substances with alkalis, has a thermometer inserted into the fused mass through the hollow shaft of the stirring apparatus. More efficient stirring is obtained than with the ordinary method of having a thermometer separate from the stirring shaft. By suitable modification, the vessel may be converted into an autoclave. L. M. Clark.

Influence of segregation on the corrosion of boiler tubes and superheaters. G. R. Woodvine and A. L. Roberts (Iron and Steel Inst., June, 1926, Advance proof. 4 pp.).—Superheater coils and tubes in high-pressure water-tube boilers fail in service much more quickly when made from segregated steel than from carefully made high-grade steel. A coil made from a solid-drawn tube of segregated steel and a tube of steel free from segregates arc-welded into one length was used as a superheater in a boiler of 200 lb. working pressure with an average steam temperature of 316—371°. After 12 months the unsegregated portion was quite sound, whereas the segregated portion, scale from the interior of which contained 0·19% of sulphur, was badly corroded and perforated. The failures of tubes made from segregated steel ingots are attributed to electrolytic action promoted by blowhole segregates. M. Cook.

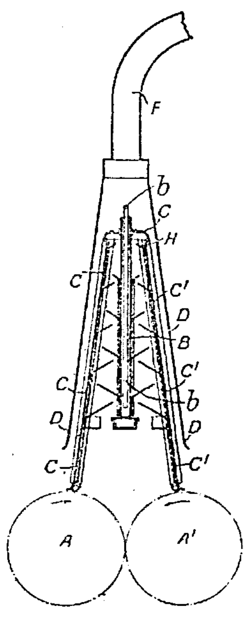
Preparation and use of industrial special sands. W. M. Weigel (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy. 14 pp.).—A description of the methods of washing and screening American sands for special purposes, *e.g.*, filter beds, abrasives, cements. In all cases the elimination of clay matter is essential and a low content of iron oxide is necessary if the product (*e.g.*, sodium silicate) is required of good colour. After washing, sands are invariably dried before grading and are despatched in the dry condition. C. A. King.

Proposed British standard table of wire screens. O. C. Ellington (J.S.C.I., 1926, 45, 128—134 t).—Screens of a standard table must be capable of easy and inexpensive manufacture in large sheets by normal trade methods and must be equally suitable for use in both manufacturing and laboratory control work. They must satisfy the following requirements :—(1) A systematic basis of graded apertures and nomenclature emphasising the primary importance of accurate aperture ; (2) the inclusion of screens equivalent in aperture to all those of established use in commerce ; (3) the use of component wires from those quoted in the British S.W.G. table ; (4) adherence to an integral and normally adopted number of meshes per linear inch ; (5) the choice of wires of the same suitable relative thickness for each screen (a value of approximately 1·5 for the ratio aperture/wire diameter, and, consequently, a value of about 36% for screening area, are suggested). A table of screens possessing these characteristics is suggested and the disadvantages and restricted scope of existing tables of screens such as the I.M.M. table of laboratory screens, are discussed.

Theories of adsorption and technique of its measurement. McBain.—See A., May, 467.

Patents.

Disintegrating apparatus. E. Roth (E.P [a] 242,312, 2.11.25, and [b] 247,526, 15.12.25. Conv., 1.11.24 and 16.2.25).—(a) Two cones rotating in opposite directions and having their bases opposed are provided on their interior surfaces with radial ribs. (b) Annular or circular ribs are also provided. B. M. Venables.

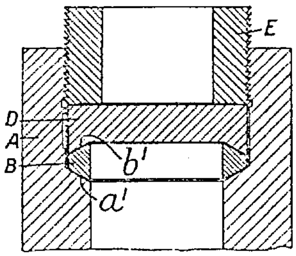


Evaporating apparatus. J. Golding and J. C. Stead (E.P. 248,131, 23.12.24).—Referring to the figure, A and A^1 are steam-heated rolls and the vapour arising therefrom is guided by deflectors against the evaporating plates, C, C^1, and the preheater, B, finally being drawn through the exhaust, F, by means of a fan. The liquid to be evaporated, *e.g.*, milk, is fed through the pipe, b, to the bottom of the preheater, B, which is in the form of a narrow deep trough, and overflows in thin sheets over the evaporating plates, C, C^1, finally being dried to a solid on the rolls, A, A^1. B. M. Venables.

Evaporators. H. Fothergill (E.P. 249,904, 1.12.24).—The liquid to be evaporated is first heated and then sprayed in the form of sheets into a vessel maintained at a pressure lower than corresponding with the b.p. of the heated liquor. The spraying apparatus consists of a tube in which longitudinal

slots are formed for the exit of liquor and which is situated under a horizontal or sloping cross baffle in the evaporating chamber. Other baffles are provided with the object of preventing entrainment, and methods of constructing the spraying tube are described. B. M. Venables.

High-pressure joints. Synthetic Ammonia & Nitrates, Ltd., and F. H. Bramwell (E.P. 249,001, 23.7.25).—In apparatus for pressures of the order of 200—400 atm., for use in the synthesis of ammonia for example, joints which may be as much as 4 ft. in



diameter are constructed as shown in the figure, where A is the body of the vessel to be sealed, B is a lens-shaped jointing ring, D a lid, and E a clamping screw. The gas-tight joint is made between the spherical surfaces of the lens ring and the conical surfaces, a^1, b^1, of the body and lid. It is essential that the lens ring be made of comparatively hard and elastic material, such as mild steel, and although it is compressed with sufficient pressure to obtain surface contact at a^1 and b^1 it must never be stressed beyond its elastic limit. The clamping screw, E, is used instead of a number of bolts in order that the clamping pressure may be applied directly over the joint surfaces, and the lid, D. is provided with a key to prevent rotation. A suitable obliquity for the conical surfaces a^1 and b^1 is stated to be 30—45°.

B. M. Venables.

Process of cleaning centrifugal bowls. Aktiebolaget Separator (E.P. 249,457, 4.3.26. Conv., 18.3.25).—Sludge is removed from the bowl of a centrifugal separator without dismantling the apparatus but after stopping it, by forcing in steam, gas, or liquid through the ordinary feed inlet and if desired also through the outlet for the lighter product, the sludge being blown out of one or both of the outlets for separated products. B. M. Venables.

Centrifugal separator. W. Birchall (U.S.P. 1,572,419—20, 9.2.26. Appl., [A] 1.4.21, [B] 10.5.21). —(A) A rotating vertical cylinder contains a rotating coaxial cylindrical screen carrying a cloth filtering medium. Within this screen rotates more rapidly a hollow shaft carrying a number of horizontal hollow arms. The mixture of solid and liquid under treatment is introduced into the hollow shaft and passes through the hollow arms to the filtering screen. The liquid passes through this while the solid accumulates on the screen and is supported in place by metal flaps attached to the arms. The arms also carry scrapers which owing to the difference in the speeds of rotation of the screen and the arms rotate on a vertical axis relatively to the screen and remove the solid material so that it falls to the bottom of the apparatus, whence it is recovered. (B) The separator is modified in that the scrapers consist of hollow cylinders fitted with slots through which the solid passes to the interior of the scraper where a vertical screw conveyor carries it to the bottom of the apparatus. Means are also provided for freeing the pores of the filtering material from solid by air under pressure. This is delivered to a compartment in the central shaft, whence it passes automatically to the outer side of those portions of the screen which are being cleared of solid by the scrapers. It passes inwards through the material thus clearing the pores. T. S. Wheeler.

Protection of metallic surfaces against incrustation and corrosion. W. Thalhofer, Assr. to A.-G. & Chem. Ind. in Liechtenstein (U.S.P. 1,558,647, 27.10.25. Appl., 14.8.23.)—This process is similar to that of E.P. 208,115 (B., 1925, 231) except that a " pulsating " electric current is used.

Recovered inorganic filter-aid. H. S. Thatcher, Assr. to Celite Co. (U.S.P. 1,571,074, 26.1.26. Appl., 24.1.23).—The spent filter-cake is dried, finely divided, and calcined at a temperature sufficiently high to remove part of the inorganic in addition to the organic impurities. H. Holmes.

Apparatus for the rapid filtration of liquids. A. Bigot (F.P. 599,161, 6.6.25).—A rotating vacuum filter drum is just completely immersed in the liquid to be filtered, and at the top is in contact with a similar drum rotating around a parallel axis outside the liquid. A greater degree of suction is maintained in the upper drum, so that the solid material collected on the lower drum passes on to the upper at the point of contact and is removed from it by a scraper. As the whole of the filtering surface of the lower drum is utilised very rapid filtration is attained.

T. S. Wheeler.

Fluid [methylene chloride] for industrial purposes [fire-extinguishing etc.] E. R. Brodton (U.S.P. 1,575,967, 9.3.26. Appl., 20.2.25).—Crude industrial methylene chloride is rectified to give a product, b.p. 38—71°, consisting chiefly of methylene chloride. It is non-explosive, does not corrode brass, copper, aluminium, or iron, and can be used as a fire extinguisher and in thermostats etc.

T. S. Wheeler.

Drying apparatus. E. C. Reader (U.S.P. 1,576,547, 16.3.26. Appl., 12.2.25).—A heating chamber communicates with an external conduit and a second conduit arranged within the chamber is provided with a frusto-conical end extending into and spaced from the end of the first conduit. Means are provided for delivering material to be dried into the first conduit and for forcing a drying fluid through the second conduit. H. Holmes.

[Magnetic] separation of gaseous or liquid mixtures. P. E. Haynes, Assr. to Linde Air Products Co. (U.S.P. 1,575,587, 2.3.26. Appl.,

2.9.19).—A mixture of fluids having different magnetic susceptibilities is passed through a conduit having a permeable wall and is acted upon by a transverse magnetic flux which draws the constituent of greater susceptibility through the permeable wall out of the main stream. B. M. Venables.

Apparatus for treating liquids with gases. W. E. Greenawalt (U.S.P. 1,579,355, 6.4.26. Appl., 11.6.23).—The gas is passed into the liquid through a hollow rotating paddle working on a vertical shaft, the fins of the paddle being so disposed that they create upward currents in the liquid. A mixture of gas and liquid is fed into the paddle and emerges into the main body of liquid through the ends and sides of the fins. A. R. Powell.

Furnace fired with coal dust and like pulverulent fuel. E. Vogt and L. Kirchhof (U.S.P. 1,580,080, 6.4.26. Appl.. 26.1.26).—A furnace is provided with a combustion chamber which is completely open to the space to be heated. The powdered fuel is introduced at the top of the combustion chamber together with primary air, and a regulated amount of secondary preheated air is also admitted through the fuel inlet. B. M. Venables.

Apparatus for taking samples from closed vacuum or pressure vessels. Farbw. vorm. Meister, Lucius, & Brüning, Assees. of G. Henninger (G.P. 423,603, 27.6.22).—An apparatus for taking samples from autoclaves or the like during working consists of a closed cylindrical container, which by rotation in a socket can be connected either to the autoclave or to the open air. Means are provided for admitting air or the like to the sampling vessel to force out the sample taken. T. S. Wheeler.

Fractional distillation and condensation of complex mixtures. Soc. Anon. d'Ougrée-Marihaye (E.P. 239,502, 20.8.25. Conv., 8.9.24).—See F.P. 598,414 ; B., 1926, 309.

Rotating retort. F. S. Bacon, Assr. to M. A. Bacon (Reissue 16,326, 20.4.26, of U.S.P. 1,556,194, 6.10.25. Appl., 22.1.26).—See B., 1925, 946.

Protecting from incrustations metallic surfaces which are in contact with water or other liquids. W. Thalhofer, Assr. to A.-G. f. Chem. Ind. in Liechtenstein (U.S.P. 1,558,646, 27.10.25. Appl., 25.10.22. Renewed 2.2.25).—See E.P. 208,115 ; B., 1925, 231.

Eliminating scale in fluid containers. K. Schnetzer, Assr. to Antiscale, Ltd. (U.S.P. 1,581,024, 13.4.26. Appl., 25.7.24).—See E.P. 208,115 ; B., 1925, 231.

Apparatus for evaporating liquids indirectly. K. L. E. Thunholm (U.S.P. 1,562,525, 24.11.25. Appl., 9.3.21).—See E.P. 156,592 ; B., 1922, 163 a.

Automatic means for letting off steam from a container having in it a boiling liquid. Farbenfabr. vorm. F. Bayer & Co., Assees. of O. Einsler (U.S.P. 1,572,272, 9.2.26. Appl., 25.11.24).—See E.P. 246,567 ; B., 1926, 304.

Centrifugal machine and process. L. D. Jones and A. U. Ayres (U.S.P. 1,575,061, 2.3.26. Appl., 29.5.25).—See E.P. 233,327 ; B.. 1926, 343.

Separating device operating by centrifugal force. S. Karpinsky and J. S. Anderson (U.S.P. 1,583,048, 4.5.26. Appl., 16.1.24).—See E.P. 214,577 ; B., 1924, 885.

Separating or recovering a gas from a mixture of gases. E. B. Miller, Assr. to Silica Gel Corp. (U.S.P. 1,577,534, 23.3.26. Appl., 15.6.22). —See E.P. 227,309 ; B., 1925, 157.

Combustion, reduction, or treatment of solid material or of pulverised minerals. X. de Spirlet (U.S.P. 1,581,397, 20.4.26. Appl., 2.10.24). —See E.P. 222,876 ; B., 1925, 871.

Straining or filtering apparatus. F. C. Fulcher (U.S.P. 1,581,998, 20.4.26. Appl., 26.11.23). —See E.P. 223,627 ; B., 1924, 1000.

Electrical purification of gases (U.S.P. 1,579,462 and G.P. 425,026, 425,039, and 425,273–4). —See XI.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Cleaning of coal. I. W. R. Chapman and R. A. Mott (Fuel, 1926, 5, 143—149).—The economy to the consumer of using clean coal is emphasised. Usually the largest lumps will contain least ash, but the actual optimum size varies from coal to coal, and practically every coal appears to produce lumps of one particular size which is the cleanest. The nature and quantity of the inorganic matter in coal, which is divided into "inherent" and "adherent" ash, is affected by the manner in which the coal-forming material was laid down and on the origin of the coal, and differs, also, in vitrain, clarain, durain, and fusain. The ash content of fusain is usually high, and, though fusain is usually present in small quantity, its great friability leads to its concentration in the fines and slurry. All cleaning operations aim at reducing the sulphur content of the coal, and the success attained will depend not so much on the total amount of sulphur present as on the relative proportions of organic sulphur, iron sulphides, and mineral sulphates. Our knowledge of the efficiency of the removal of phosphorus in different washing processes is meagre, but it appears that under the most favourable conditions the percentage removal is small. Salt can be only partially removed from most coals by washing, and it is important to avoid an accumulation of soluble salts in the washing water. The fusibility of the coal ash may be markedly influenced by washing, in so far as the relative amounts of iron, lime, magnesia, silica, and alumina are thereby altered. W. T. K. Braunholtz.

Application of the Chance sand flotation process to washing bituminous coal. A. Greenwell (Fuel, 1926, 5, 163—166).—A detailed description of an American plant in which 335 tons per hr.

of bituminous coal are washed in conical washers by the Chance sand–water process (cf. B., 1924, 699). The material treated is sized to pass through a screen with $4\frac{1}{2}$ in. sq. openings and over a $\frac{3}{8}$ in. screen, the washing of the smalls being considered unnecessary. The refuse is drawn off at the base of the cone, and clean coal, sand, and water overflow at the top. The sand is separated from both the coal and refuse by screening, and is returned to the washer. The ash in the washed coal is constant within about 0·75% and the sulphur within 0·2%.
W. T. K. BRAUNHOLTZ.

Rate of travel of fusion zones in coke ovens. W. P. RYAN (Fuel, 1926, **5**, 150—157).—Curves are drawn illustrating the changes in temperature at various points in an oven during carbonisation, the distribution of temperature across the oven at any moment, and the passage across the oven of two isotherms lying in the neighbourhood of the fusion point and of the point of maximum pressure. It is shown that if maximum pressure is not synchronous with fusion it follows shortly after, and that a given condition of temperature or pressure moves across the oven at a variable rate depending on the distance from the heating wall. The thermal conductivity of coal does not change in order of magnitude on fusion and conversion into semi-coke, but that of coke is much higher than that of coal. The heat of fusion and thermal decomposition is negligible in effect on the rate of heating of the coal. The rate of travel of the fusion zone is rapid nearest the heating walls, decreasing to a minimum as the fusion zone passes towards the centre of the oven, and finally increasing again as it comes near the centre, where the coal comes under the influence of heat transmitted from both sides.
W. T. K. BRAUNHOLTZ.

Thermal decomposition of cellulose and lignin in presence of catalysts and hydrogen under pressure. A. R. BOWEN and A. W. NASH (Fuel, 1926, **5**, 138—142; cf. J.S.C.I., 1925, 507 T).—When cellulose and lignin are gradually heated to about 450° with hydrogen under pressure (maximum up to about 240 atm.) in the presence of catalysts (*e.g.*, alumina and nickel oxide) liquid and gaseous products are formed, but the oils obtained are not fully saturated, owing probably to the deposition of small quantities of coke on the catalyst. Cellulose semi-coke (prepared at 400°) can also be hydrogenated and yields an oil having a relatively low proportion of volatile constituents. The extent of conversion is greater with cellulose than with lignin or cellulose semi-coke, but phenols are formed to the greatest extent (4·8%) from lignin, which is also characterised by the formation of substances soluble in phenol. The volatile oil fractions have in each case an appreciable hydrocarbon content, the saturated portion of which, in the case of cellulose and cellulose semi-coke, has a paraffinic odour.
W. T. K. BRAUNHOLTZ.

Corrosion in gas meters. F. FIRTH (Gas J., 1926, **174**, 41—45).—Experiments on the corrosion of iron in electrolytes seemed to show that the rate and extent of corrosion are determined not so much by the concentration of dissolved salts as by the concentration of oxygen in contact with the wetted iron. To prevent corrosion it is recommended that the oxygen content of the gas be reduced by passing over easily corrodible iron turnings in purifier boxes, and that the gas be dried by refrigeration, by compression, or by the use of chemical desiccating agents.
S. PEXTON.

New forms of gas analysis apparatus. E. OTT (Gas u. Wasserf., 1926, **69**, 289—295).—An apparatus for accurate gas analysis consists of a burette and compensator, absorption pipettes, and a quartz tube containing copper oxide or a capillary packed with fine platinum wire. The compensator and burette are connected by a manometer which is always adjusted before taking a reading; the confining liquid is mercury; the free spaces in the apparatus are filled with nitrogen. Carbon dioxide is determined by absorption in caustic alkali solution; unsaturated hydrocarbons in oleum (the oleum absorption pipette, it was found, is best filled with glass rods; the diminution in volume of the absorbent reduces the risk of error due to solution of the gas, etc.); oxygen in pyrogallol solution; and carbon monoxide in ammoniacal cuprous chloride. Hydrogen is burnt at 300° over copper oxide. Carbon monoxide may be estimated by this method alternatively; but in this case combustion should take place over platinum to avoid the absorption of the resulting carbon dioxide (especially if its partial pressure is high) by the copper and copper oxide. Allowance for volume changes due to the reduction of copper oxide to copper, and other intricacies in this combustion process are discussed. Methane is burnt at a bright red heat over copper oxide, or if ethane is present, over platinum, oxygen being admixed. Figures from actual analyses are given, and the method of calculating results is shown. For technical purposes a portable type of apparatus is described; it is somewhat modified and does not possess a compensator; in this form it is an elaboration of the Orsat.
R. A. A. TAYLOR.

Grosny petroleum. A. SACHANOV (Neftjanoe slancevoe Chozjajstvo, 1925, **9**, 554—563; Chem. Zentr., 1926, I. 2269).—The oil from the newer Grosny oilfields contains 2·5—4·7% of paraffin wax, whilst that of the older may be divided into two types containing 2—2·5% and less than 0·4% of paraffin wax respectively. A typical oil of the former class had η^{20} 0·098 (C.G.S. units), *d* 0·838, f.p. + 11°, and contained 12% of resins. On distillation it gave 7·64% below 100°, 15·67% at 100—200°, 13·31% at 200—270°, and 62·75% of residue having η^{50} 3·6 (Engler), *d*, 0·898, f.p. 30°. Distillation of this residue with steam gave 25·4% (of the petroleum) of solar oil, 10·2% of spindle oil, 10·2% of machine oil, 12·2% of "goudron" oil, 4·5% of resin, and 0·9% of asphaltenes. An oil of the second type had η^{20} 0·075, *d* 0·851, f.p. —15° to —20°, and contained 15% of resins. It yielded on distillation 7·73%

below 100°, 20·06% at 100—200°, 12·54% at 200—270°, and 59% of residue having η^{50} 4·5, *d* 0·909, f.p. 19°, and giving on distillation with steam 23·6% of solar oil, 7·1% of spindle oil, 7·1% of machine oil, 13·9% of "goudron" oil, 6·5% of resin, and 1·5% of asphaltenes. An oil of the third type had η^{20} 0·140, *d* 0·879, f.p. below —20°, and contained 20% of resins. When distilled alone or with steam it gave 8·46% below 100°, 20·07% at 100—200°, 12·55% at 200—270°, 14·1% of solar oil, 9·4% of spindle oil, 9·4% of machine oil, 4·7% of cylinder oil, 11·2% of "goudron" oil, 0·2% of paraffin wax, 8% of resin, and 2% of asphaltenes. This type of paraffin-free oil is characterised by its high asphaltene content. A fourth type of product of the Grosny oilfields is natural gas, which has a high density and benzine content owing to the high temperature (up to 90°) of the majority of the boreholes. The gas comprises about 30—40% of condensable gases and 60—70% of methane, ethane, and propane. The benzine obtained from the natural gas has *d* 0·677, and 58% distils below 100°. W. T. K. Braunholtz.

Abridged volume correction table for petroleum oils (U.S. Bur. Standards, Suppl. to Circ. 154, 8.10.25, 4 pp.).—The abridged table is for use in converting oil volumes at various temperatures to the volume at 60°F. (15·5°) when extreme accuracy is not required. For high accuracy the table in Circular 154 should be used. H. Moore.

Synthesis of petroleum at atmospheric pressure from products of coal gasification. F. Fischer and H. Tropsch (Brennstoff-Chem., 1926, 7, 97—104; Ber., 1926, 59, 830—836, 923—925).—Petroleum products, from benzine to paraffin wax, can be synthesised at atmospheric pressure from gas mixtures containing carbon monoxide and hydrogen (*e.g.*, water-gas) by passing the gas over a catalyst at moderate temperatures (up to about 270°). Finely-divided metals of the 8th group of the periodic system, prepared by reducing their oxides with pure hydrogen at 350°, may be used as catalysts, alone or in conjunction with metal oxides (*e.g.*, oxides of chromium, zinc, beryllium, etc.), and with or without a carrier (*e.g.*, pumice, asbestos). With iron alone the reaction is slow; cobalt is more active, but with nickel the main product is methane, which is also the sole product in all cases at higher temperatures. Increasing the basicity of the catalyst by addition of alkali carbonates or hydroxides increases the proportion of higher-boiling products. It is essential that the gases used should be free from sulphur compounds, which poison the catalyst, and it may be necessary from time to time to free the latter from clogging by heavy paraffins. As an example of the yields obtained, 100 g. of liquid and solid products were recovered by repeatedly circulating 1 cb. m. of water-gas over an iron-copper catalyst. The products included "gasol" (ethane, propane, butane, with only traces of olefines), benzine, "petroleum," and paraffin wax. The benzine, extracted from the gases by active charcoal, was water-white and remained so, and had d^{15} 0·6718, cal. val. (gross) 11,360 cals. per g., and spontaneous ignition temperature 335°. On distillation it gave 18·2% at 50°, 75·3% at 100°, 92·3% at 150°. The "petroleum" had d^{20} 0·7804, and yielded 4·4% at 180°, 14·4% at 200°, 49·2% at 250°, 70·4% at 300°, and 83·2% at 330°. The paraffin wax, recrystallised from acetone, was colourless and odourless, and had f.p. 61°. The catalyst is supposed to function by virtue of the intermediate formation of unstable metal carbides of high carbon content. W. T. K. Braunholtz.

Action of ozone upon petroleum. I. Action of ozone on the saturated hydrocarbons of petroleum. G. Kita, R. Abe, and S. Tada (J. Soc. Chem. Ind. Japan, 1926, 29, 7—12).—By the action of ozonised oxygen containing about 5% of ozone on a fraction (b.p. 150—300°, mainly saturated hydrocarbons) of Kurokawa petroleum, the iodine and acid values, sp. gr., refractive index, and viscosity increased to an extent depending on the time of the treatment, while the rotatory power decreased. In the case of liquid paraffin (Jap. Pharm. IV.), the acid value, sp. gr., and viscosity increased slightly, while the rotatory power decreased and the iodine value and refractive index were not influenced by treatment with the ozonised oxygen. K. Kashima.

Methods of testing oils for transformers, switches, and turbines. G. Baum (Z. angew. Chem., 1926, 39, 473—475).—A review of the most important methods for testing the suitability of an oil for use as an electrical insulator, pointing out the unsatisfactory results given by most methods and emphasising the need of further research to discover the reason why the insulating power of certain oils breaks down more quickly than that of others. Comparable results for the amount of tarry matter formed by oxidation of the oil are not obtained by any of the present methods—sodium peroxide, exposure to air at 120°, and bubbling air through the oil at 120°—and considerable difference of opinion exists as to the value of the method involving agitation with sulphuric acid. A. R. Powell.

Syzran asphalts. A. Parschin (Neftjanoe slancevoe Chozjajstvo, 1925, 9, 582—584; Chem. Zentr., 1926, I, 2270).—The bitumen is separated from the bituminous rock by boiling with acidulated water. A type of bituminous sandstone known as "Garj" yields no bitumen by this process and is regarded as a waste product, although it contains as much as 5·9—16·8% of bitumen that can be extracted with chloroform. The bitumen is not separated by boiling water owing to its high m.p. (96—147°). A sample having m.p. 107° contained 52·7% of asphaltenes (*d* 1·074), 29·0% of resins (*d* 1·069), and 16% of oils (*d* 0·910). The extraction of the bitumen with benzene or carbon disulphide could probably be applied commercially. W. T. K. Braunholtz.

Solubility of formolites. K. Kostrin (Neftjanoe slancevoe Chozjajstvo, 1925, 9, 579—581; Chem. Zentr., 1926, I, 2270—2271).—By shaking

one of the oils obtained as a by-product in the refining of machine oil with concentrated sulphuric acid and 40% formaldehyde solution, warming, diluting, and neutralising, a solid formolite was obtained. This was extracted successively with benzene, alcohol, carbon tetrachloride, chloroform, acetone, and ether. The quantity of formolite soluble in benzene and of that insoluble in all solvents increased with the b.p. of the original oil.

W. T. K. BRAUNHOLTZ.

Naphthenic acids derived from kerosene distillate of Californian petroleum. Y. TANAKA and S. NAGAI (J. Soc. Chem. Ind. Japan, 1926, 29, 58—63).—From the waste lye produced in the refining of kerosene distillate 0·19% (on the weight of kerosene) of crude naphthenic acids was obtained and these were purified by conversion into the methyl esters. About 73·5% of the esters distilled at 120—180°/8·9—9·0 mm. On saponification of the methyl esters a mixture of pure naphthenic acids was obtained, d_4^{20} 0·9448, n_D^{20} 1·4661, and neutralisation value 246·2. About 77% of the mixture distilled at 160—230°/8·9—9·0 mm. The main fractions were considerably lighter (d 0·95) than those (d 0·98) of the naphthenic acids obtained from a gas oil distillate (B., 1926, 307). A white crystalline mass separated in the high-boiling fractions.

K. KASHIMA.

Micro-structure of coal. R. THIESSEN (J. Soc. Arts, 1926, 74, 535—557).

Occurrence of ulmic compounds in deteriorated fabrics and bearing of their formation on the origin of peat and coal. A. C. THAYSEN, W. E. BAKES, and H. J. BUNKER (Fuel, 1926, 5, 217—220).—See B., 1926, 305.

Determination of olefinic, aromatic, and saturated hydrocarbons in petroleum and tar oil fractions. N. DANAILA and E. MELINESCU (Bul. Chim., Soc. Româna Stiin., 1924, 27, 3—10).—See B., 1925, 835.

Utilisation of aromatic hydrocarbons in petroleum fractions. I. Preparation of α-trinitrotoluene. N. DANAILA and N. POPA (Bul. Chim., Soc. Româna Stiin., 1924, 27, 45—80).—See B., 1925, 745.

See also A., May, 464, **System ethyl alcohol-benzene-water** (BARBAUDY). 467, **Adsorption of ethylene and hydrogen by zinc oxide, iron oxide, nickel, and copper** (LAZIER and ADKINS). 480, **Combustion of well-dried carbon monoxide and oxygen mixtures** (BONE and WESTON; BONE, FRASER, and NEWITT).

Silica brick from coke-oven walls. COLE.—See VIII.

PATENTS.

Coal washeries. A. ROBINSON, and SIMON-CARVES, LTD. (E.P. 249,977, 10.2.25).—The fine coal and slurry are independently separated from the coarse coal and treated for the removal of water. The sieved fines are allowed to drain in bunkers and the water recovered can be utilised again in the washery. The slurry is jigged on a sieve. Both products can then be mixed with the coarse coal, from which the water easily drains when the slurry and fines are absent.

S. PEXTON.

Decomposition of peat. G. MEUNIER and C. DE BRISSON DE LAROCHE (F.P. 597,387, 24.4.25).—Colloidal peat is treated with alkalis to separate fibrous material and other fine constituents, and the latter are hydrolysed to recover the humus constituents.

L. A. COLES.

Process of modifying ignition temperature etc. of carbonaceous materials. I. H. DERBY and C. B. EDWARDS, Assrs. to P. C. REILLY (U.S.P. 1,576,179, 9.3.26. Appl., 11.8.23).—The ignition temperature of coke or similar substance is lowered some 200° and its rate of burning correspondingly raised by coating it with lead, lead oxide, etc., or with a thallium compound, copper oxide, barium peroxide, or generally with a compound of one of these metals which easily yields the oxide on heating or with the oxide itself. For example, 42,000 pts. of coal tar are mixed with 160 pts. of lead oxide and distilled. The coke obtained is as easily ignited as charcoal and has a lower ash content. The products described are of value as a covering for the molten lead used in the heat treatment of steel; owing to their freedom from ash and relatively high sp. gr. they are superior to charcoal, which is usually employed for this purpose.

T. S. WHEELER.

Coke ovens and batteries thereof. KOPPERS CO., Assees. of J. BECKER (E.P. 232,173, 24.12.24. Conv., 10.4.24).—The battery units have two pairs of heating walls with vertical flues and connected by a number of cross-over ducts at both the ram and quencher sides so that the gases flow in the two sides in separate paths without intermingling, the direction of flow in one pair of heating walls being the reverse of that in the other. Reversible regenerators are arranged in a row beneath the oven, the arrangement being such that the fuel gas regenerators are separated by air regenerators from the regenerators receiving waste gases from the downflow heating walls.

W. T. K. BRAUNHOLTZ.

Coking retort ovens. KOPPERS CO., Assees. of J. VAN ACKEREN (E.P. 246,500, 29.10.24. Conv., 4.1.24).—In a battery of regenerative vertical retort ovens. the long vertical flues are supplied with preheated air and fuel gases both at the top and middle. The heating flues are interconnected in pairs at the bottom by ducts which pass around the end of the retorts on the regenerator side. Half the combustion flues operate for inflow, whilst the the other half operate for outflow. During the reversal periods some retorts are heated from both sides by down-flowing flame gases, but as the reversals are regular all retorts get substantially the same treatment. The regenerators are so grouped that those containing fuel gas are separated

from those containing waste gases by secondary air regenerators. S. Pexton.

Horizontal coke oven. H. B. Carpenter (U.S.P. 1,579,075, 30.3.26. Appl., 18.10.21).—The apparatus consists of retorts and flues arranged alternately and separated by common walls. The sidewalls of the lower halves of the retorts and of the flues are parallel. The sidewalls of the upper half of each retort slope slightly towards the central vertical plane of the retort. The sidewalls of the upper half of each flue slope away from the central vertical plane of the flue. The retorts are thus narrower at the top than at the bottom, while with the flues the reverse is the case. R. A. A. Taylor.

Stand-pipes for use in the manufacture of coal gas. J. G. W. Aldridge, W. M. Carr, and T. J. Ashley (E.P. 248,557, 3.3.25).—The stand-pipes carrying the crude gases from a tier of retorts up to the foul main increase in cross-sectional area in the direction of flow of the gases, thereby reducing the gas velocity and increasing the efficiency of the cooling and scrubbing effected by a stream of liquid which is projected into the upper end of the stand-pipe, passes down counter-current to the gas, and is drawn off at a seal-box at the level of the stage floor. The connexions from the retort to the stand-pipe are provided with valves and means for deflecting the stream of liquor from the connexion. The upper portion of the stand-pipe may be further extended and enlarged above the retorts, and provided with baffles for improving the contact between the liquor and gas. A. B. Manning.

Manufacture of hard coke. P. Dvorkovitz (E.P. 249,901, 3.11.24).—Coal is subjected to a preliminary carbonisation at 500° prior to carbonising at 900°, with a view to increase considerably the output of the high-temperature carbonising plant. In addition, non-oxidising gases containing hydrogen are passed through the coke at 700—800°, whereby sulphur and nitrogen are eliminated from the coke. S. Pexton.

Treatment of wood. W. A. Audibert (F.P. 586,856, 4.10.24).—Wood is de-aerated in closed vessels, treated with acids or acid salts under pressure, and washed with water. The treatment results in the decomposition of the ligneous constituents, and the destruction of material capable of causing rot, and also the production or separation of resin, acetone, acetic acid, etc., which are recovered. L. A. Coles.

Manufacture of [water-]gas. Humphreys and Glasgow, and J. C. Stelfox (E.P. 246,970, 16.12.24).—In a water-gas producer the fuel is coked by contact with water-gas and excess steam during the run with superheated steam, and by contact with water-gas obtained during a run with steam which has not passed through the recuperator, with or without part of the coal gas and water-gas mixed and superheated with fresh steam. The blow may be prolonged after heating the recuperators, and the resulting producer gas used to coke the fuel and then conveyed to storage. The producer, *a*, is connected by pipes, *d*, *e*, *f*, with a carburettor, *b*, and pipes, *g*, *h*, lead to storage. The recuperator, *c*,

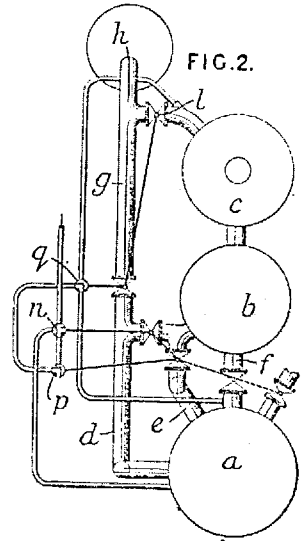


FIG. 2.

is connected to the pipe, *h*, through a valve, *l*, to enable water-gas to be re-circulated through the plant. Steam is supplied through valves, *n*, *p*, *q*, to the required parts of the plant, and the valves are coupled as shown by the full thick lines; the broken lines indicate interlocking means between the valves. S. Pexton.

Producing a high-grade mixed gas. C. Asbeck (E.P. 249,772, 6.11.25).—The plant comprises a gas producer and an auxiliary coal distillation retort. The producer, which is an elongated water-gas generator, is fed with fresh coal which is distilled in the upper part by the sensible heat of the water-gas which streams through it during the "run." During the blow the waste gases are led off from the generator below the coal distillation zone and burnt in a chequered chamber which surrounds the auxiliary retort in which coal is carbonised and from which coke is discharged. The waste gases from the chequered flues are passed through a waste-heat boiler which is fed with water preheated by circulating through an annular space surrounding the lower part of the auxiliary retort, thereby cooling the coke. The distillation gases from the producer and the retort during the "run" are circulated through the hot chequered chamber surrounding the retort in order to crack the tarry vapours and completely fix them. S. Pexton.

Recovery of carbon monoxide from industrial gases. A. A. L. J. Damiens (G.P. 425,418, 26.11.24. Conv., 12.4.24. Addn. to 422,621).—Sulphuric acid of *d* below 1·84 is used in the process described in the chief patent (cf. E.P. 230,106; B., 1925, 847), and temperatures up to or above 100°, according to the dilution of the acid, are employed, the gases being compressed during the absorption, if necessary. The cuprous compound and carbon monoxide are

recovered by heating the complex compound which is formed, with sulphuric acid of *d* below 1·84.
L. A. Coles.

Treating tar water. Badische Anilin- & Soda-Fabr., Assees. of C. Schneider (U.S.P. 1,579,957, 6.4.26. Appl., 7.11.25).—The hot tar water is treated with an alkali hydroxide and exposed to air. It is then used again for cooling coal gas or the like.
C. Irwin.

Blending and purifying light hydrocarbons. G. Egloff, Assr. to Universal Oil Products Co. (U.S.P. 1,575,905, 9.3.26. Appl., 31.12.20).—Gasoline-like hydrocarbons admixed with uncondensable gas are blended with a heavier absorbing oil and purified by passing the mixture through a solution having a base of caustic soda and litharge. H. Moore.

Oil distillation. J. E. Bell Assr. to Sinclair Refining Co. (U.S.P. 1,576,401, 9.3.26. Appl., 7.10.21).—Oil is fractionally distilled by passing through a series of stills at successively higher temperatures. Part of the vapour from each still is condensed by oil at a lower temperature, and the oil in intermediate stills is heated by vapours from stills later in the series.
H. Moore.

Heating system for oil stills. F. E. Wellman, Assr. to Kansas City Gasoline Co. (U.S.P. 1,576,563, 16.3.26. Appl., 17.12.21).—A vertical still is surrounded by an annular combustion space, the upper part of which communicates with the stack. Means are provided for producing localised combustion in the lower part of the space, and for constantly changing the point of application of the resulting flame to the still wall.
H. Moore.

Continuous process for refining petroleum and the like. H. L. Carroll (F.P. 598,295, 2.9.24). —Crude petroleum, or petroleum from which the lighter constituents have been removed, is circulated rapidly through a vessel in which it is heated to a temperature approximating to the boiling point of the highest-boiling fractions to be distilled off, after which it enters the lower end of a column in which the escaping vapours are subjected to fractional condensation.
L. A. Coles.

Breaking [petroleum] emulsions. L. G. Gates, Assr. to Standard Oil Co. (U.S.P. 1,573,389, 16.2.26. Appl., 14.5.25).—A petroleum emulsion of the water-in-oil type is brought into contact with cotton, iron, monel metal, or other suitable substances whereby the positively charged oil acquires a negative charge and the repulsion between the acquired charge and the similar charge on the water particles breaks the emulsion. The crude oil emulsion heated to about 70° is delivered to one side of a filter screen in which the filtering medium is cotton, and is withdrawn from the other side; preferably several filter units in parallel are used. To prevent clogging of the pores of the cotton the direction of flow of the oil is frequently reversed. The filtering medium is earthed to remove the positive charge which it acquires during the process. The rate of separation of oil and water is increased if a cloth filter be fitted to the pipe discharging the mixture from the filter into the separating tank.
T. S. Wheeler.

Emulsified solid greases. M. J. Heitmann (E.P. 232,259, 14.4.25. Conv., 10.4.24).—An emulsified solid grease, containing about 50% of water, particularly suitable for lubricating axles of trucks etc., as its consistency is maintained irrespective of temperature changes, is obtained by subjecting lubricating oil to a preliminary treatment by electric glow discharges (cf. E.P. 209,060) before adding the water. No soap or soap-forming substance is used.
H. M. Langton.

Making organic-acid soap [cutting oil] from mineral oils. E. M. Hughes, Assr. to Sun Oil Co. (U.S.P., 1,577,723, 23.3.26. Appl., 2.7.18).—Lubricating oil stock from the distillation of crude petroleum is agitated with sulphuric acid and the sludge drawn off. The oil is then agitated with caustic soda. An emulsion consisting of soap, salt water, free alkali, and unsaponifiable mineral oil is removed, after which the soap is decomposed with a dilute solution of sulphuric acid. Water and salts are separated and the remaining product is saponified and mixed with a mineral oil. A cutting oil is thus formed which gives a milky emulsion when mixed with water.
R. B. Clarke.

Separating [paraffin] wax from lubricating oil. E. Petty, Assr. to De Laval Separator Co. (U.S.P., 1,577,852, 23.3.26. Appl., 24.8.21).—Lubricating oil stock from crude petroleum is mixed with a light petroleum distillate and agitated with sulphuric acid. After removing the sludge, the oil is washed with water, neutralised by agitating with caustic soda, and again washed. Soap and alkaline water are removed in a centrifugal separator, after which the oil is clarified and decolorised by stirring with fuller's earth and filtering on a vacuum filter. Clarification is completed in a centrifugal clarifier, Wax is precipitated from the oil by chilling it, partly with brine and partly with cold oil. Oil and wax are then separated by centrifuging.
R. B. Clarke.

Treating gilsonite. Gilsonite products. Gilsonitic product and its manufacture. (A, B) C. N. Forrest, and (C) C. N. Forrest, H. P. Hayden, and O. R. Douthett, Assrs. to Barber Asphalt Co. (U.S.P. 1,573,764–5, 16.2.26, and 1,578,235, 23.3.26. Appl., [A] 18.9.19, [B] 19.9.22, [C] 28.8.19).—(A, B) Gilsonite is distilled to about 450°, care being taken as the temperature approaches 285° when an exothermal reaction occurs. The products consist of fuel gas (12·75%), consisting chiefly of methane, and carrying light oil (2%), and ammonia (0·25%), an oil distillate (55%), and coke (30%). The coke contains 5—6% of nitrogen, which is recovered as ammonia when the coke is gasified in a producer. The oil distillate is fractionated, preferably in a current of carbon dioxide, as described in U.S.P. 877,620 (cf. F.P. 379,521; B., 1907, 1233) and yields a light solvent oil, an

intermediate gas oil, and a heavy oil which gives a lubricating oil and wax. All these oils have both asphaltic and paraffinoid characteristics, contain a high proportion of nitrogen, and are highly unsaturated. (c) The middle oil is purified by treatment with sulphuric acid and is then sulphonated with mixtures of oleum and sulphuric acid containing increasing proportions of oleum. A thin clear oil with the properties of a highly refined petroleum oil remains. The acid sludges on dilution yield three layers, a semi-drying oil, a solution of water-soluble sulphonic acids, and dilute sulphuric acid. The sulphonic acid solution can be used for the hydrolysis of fats, and the salts of these acids have detergent properties. T. S. Wheeler.

Briquetting fuels. R. Kattner, Assr. to Frankensteiner Magnesitwerke A.-G. (U.S.P. 1,562,564, 24.11.25. Appl., 19.9.25).—See E.P. 241,175; B., 1926, 4.

Gas producer with a revolving grate. W. Bennhold, Assr. to J. Pintsch A.-G. (U.S.P. 1,581,159, 20.4 26. Appl., 18.8.20).—See E.P. 146,964; B., 1921, 803 a.

Producing hydrogen from water-gas. J. H. de Graer (U.S.P. 1,583,673, 4.5.26. Appl., 26.1.23). —See E.P. 212,943; B., 1925, 356.

Manufacturing active carbon. Badische Anilin- & Soda-Fabr., Assees. of F. Winkler (U.S.P. 1,582,718, 27.4.26. Appl., 29.9.23).—See E.P. 216,761; B., 1924, 664.

Oil still. J. Primrose, Assr. to Power Specialty Co. (U.S.P. 1,566,921, 22.12.25. Appl., 19.6.20).— See E.P. 176,101; B., 1922, 284 a.

Fractionation of hydrocarbon oils and like liquids. R. H. Crozier (U.S.P. 1,583,340, 4.5.26. Appl., 7.7.25).—See E.P. 240,502; B., 1925, 951.

[Bituminous] emulsion. L. Kirschbraun (Reissue 16,328, 20.4.26, of U.S.P. 1,479,042, 1.1.24. Appl., 30.12.25).—See B., 1924, 210.

Bituminous emulsion. G. S. Hay, Assr. to Asphalt Cold Mix (1925), Ltd. (U.S.P. 1,582,467, 27.4.26. Appl., 18.8.25).—See E.P. 243,398; B., 1926, 120.

Apparatus for discharging the solid products from retorts and the like. W. R. Degenhardt (E.P. 250,716, 23.2.25).

See also Class III., **Purifying petroleum oils** (E.P. 226,188); **Paraffin wax from low-temperature tar** (F.P. 597,919). Page 486, **Making hydrocarbons and cyanides** (U.S.P 1,567,241). 487, **Hydrogen** (E.P. 249,925). 488, **Sulphur from gases** (G.P. 425,664).

III.—TAR AND TAR PRODUCTS.

Patents.

Purifying commercial benzene [and petroleum oils]. Schlesisches Kohlenforschungs-Inst. der Kaiser-Wilhelm-Ges. (E.P. 226,188, 27.11.24. Conv., 11.12.23).—Crude benzol or other distillates of coal, lignite, or petroleum are purified by heating to 100—250° under a pressure of 100 atm. or to 250—500° at atmospheric pressure. Unsaturated constituents unstable to light are polymerised and carbon disulphide and hydrogen cyanide are destroyed, but thiophen is not affected. The residues of the subsequent distillation of the treated oil are lightened in colour by the presence of copper, tin, or lead, *e.g.*, as lining to the apparatus used. (Reference is directed in pursuance of Sect. 7 (4) of the Patents and Designs Acts, 1907 and 1919, to E.P. 223,066 and 123,806.) C. Irwin.

Recovery of paraffin wax from low-temperature tar. A. Mailhe (F.P. 597,919, 13.8.24). —The tar is treated cold with methyl alcohol, and undissolved wax is filtered off, or it is treated with the alcohol at 40—60°, allowed to cool until no more wax separates, and is then filtered. The solution is distilled to recover the methyl alcohol. L. A. Coles.

Extracting benzols from heavy oils. E. A. Barbet (U.S.P. 1,561,899, 17.11.25. Appl., 21.3.21). —See F.P. 473,082; B., 1915, 485.

Producing viscous liquids [from tar oils]. L. Lilienfeld (U.S.P. 1,563,203, 24.11.25. Appl., 10.1.21).—See E.P. 149,317; B., 1921, 840 a.

IV.—DYESTUFFS AND INTERMEDIATES.

Colour measurement and standardisation. L. C. Martin (J. Oil and Colour Chem. Assoc., 1926, 9, 92—105).

Fusion pot. Babasinian and Billinger.— See I.

Patents.

Manufacture [purification] of reddish-yellow vat dyestuffs [flavanthrone]. Brit. Dyestuffs Corp., J. Baddiley, and A. Shepherdson (E.P. 249,993, 5.3.25).—Crude flavanthrone is purified by fractional precipitation from its solution in concentrated sulphuric acid by dilution with water, or by treatment with sulphuric acid of such concentration that the impurities dissolve but not the flavanthrone. For example, 15 pts. of ice are added to a solution of 10 pts. of crude flavanthrone (Indanthrene Yellow G) in 100 pts. of 100% sulphuric acid at 30—40°, and, after stirring for 2 hrs., the precipitated dye is filtered off and washed successively with 80—90% sulphuric acid and water, yielding a product giving shades as pure as those obtained from Indanthrene Yellow R. A dye yielding dull shades is precipitated on further dilution of the sulphuric acid after filtration. L. A. Coles.

[Vat] dyestuffs. Soc. of Chem. Ind. in Basle, Assees. of J. Frölich (U.S.P. 1,575,678–9, 9.3.26. Appl., [a] 21.10.24, [b] 7.11.24).—2 : 5-Diarylido-*p*-benzoquinones are treated with sulphur dichloride, if necessary in presence of a diluent such as nitrobenzene, and with addition of an alkaline compound

and a condensing agent, such as sulphuric acid, to yield vat dyestuffs which may contain both sulphur and halogen and which give fast shades on animal fibres, *e.g.*, wool, darker shades being obtained at higher temperatures. Particularly suitable are 2 : 5-diarylidobenzoquinones not substituted or only monosubstituted in the 3- or 6-position in the quinone nucleus, for such compounds react at ordinary temperatures, whereas 3 : 6-disubstituted compounds require a higher temperature. The 2 : 5-diarylido-compounds are obtained from two aromatic amines which may be the same or different and which may be monosubstituted at the nitrogen atom and mono- or poly-substituted in the nucleus by halogen, or alkoxy-, alkyl-, aryloxy-, nitro-, amino-groups, etc. The shades obtained vary from yellow-brown to red-brown, olive green, green-blue, and grey. For example, 14·5 pts. of 2 : 5-dianilidobenzoquinone are treated for a long period in nitrobenzene at 5° with 11·3 pts. of sulphur dichloride to give a dyestuff yielding blackish-brown shades on wool in an alkaline hyposulphite vat. If 17 pts. of sulphur dichloride are used the product gives full brown shades when the condensation is carried out at 5°; the products obtained at 55°, 105°, and at the boiling point of nitrobenzene, give dark brown, red-brown, and grey to black shades respectively. 90 pts. of 3 : 6-dichloro-2 : 5-dianilidobenzoquinone treated with 78 pts. of sulphur dichloride in nitrobenzene at its boiling point yield a product giving blue-grey shades. (B) If in the processes described above sulphur dichloride is replaced by sulphur monochloride, the temperature being maintained below 100°, products are obtained giving in an alkaline hyposulphite vat fast yellow-brown to red-brown, olive green, green, and grey tints on wool. For example, 7·2 pts. of 2 : 5-dianilidobenzoquinone are treated for a long period in nitrobenzene with 11 pts. of sulphur monochloride at 2—6° to yield a product giving fast brown shades on wool; if the reaction is carried out in presence of sodium acetate the shades are olive-brown, whilst with magnesium oxide a product giving dark brown shades is obtained. 14·5 pts. of 2 : 5-dianilidobenzoquinone and 34 pts. of sulphur monochloride heated in sulphuric acid at 70° yield products giving yellow-green to green shades according to the time of reaction. 40 pts. of 6-chloro-2 : 5-dianilidobenzoquinone treated at 5° in nitrobenzene with 50·5 pts. of sulphur monochloride yield a product which gives fast yellow-brown shades. T. S. WHEELER.

Manufacture of oxidation products of aromatic hydrocarbons [phthalic acid, anthraquinone] by air oxidation. A. G. GREEN (E.P. 249,973, 6.2.25).—In the oxidation, *e.g.*, of naphthalene to phthalic acid or of anthracene to anthraquinone, the catalyst, consisting of pumice stone coated with vanadium pentoxide, is disposed upon perforated plates in a reaction chamber, and the temperature during the reaction is regulated by introducing an atomised spray of water into the spaces between the plates. L. A. COLES.

Manufacture of vat-dyestuffs. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 245,623, 16.2.25).—See F.P. 593,117; B., 1926, 266.

Indigoid dyestuffs. SOC. OF CHEM. IND. IN BASLE, Assees. of B. MAYER and J. WÜRGLER (U.S.P. 1,561,560, 17.11.25. Appl., 4.1.24).—See E.P. 210,413; B., 1925, 66.

Manufacture of vat [dibenzanthrone] dyestuffs. BADISCHE ANILIN- & SODA-FABR., Assees. of H. NERESHEIMER (U.S.P. 1,562,468, 24.11.25. Appl., 11.11.24).—See E.P. 212,145; B., 1924, 369.

Black [dibenzanthrone] vat dyestuff composition. BADISCHE ANILIN- & SODA-FABR., Assees. of R. JUST, K. WILKE, and P. NAWIASKY (U.S.P. 1,581,638, 20.4.26. Appl., 26.1.25).—See E.P. 241,437; B., 1925, 983.

Acid azo dyestuffs for wool containing chromium. SOC. OF CHEM. IND. IN BASLE, Assees. of H. FRITZSCHE, H. GUBLER, and F. STRAUB (U.S.P. 1,581,572, 20.4.26. Appl., 15.10.23).—See E.P. 210,890; B., 1924, 328.

Azo dyestuffs. SOC. OF CHEM. IND. IN BASLE, Assees. of G. DE MONTMOLLIN and G. BONHÔTE (U.S.P. 1,582,029, 27.4.26. Appl., 23.3.25).—See E.P. 232,251; B., 1925, 910.

Hydrogenation of naphthalene. G. SCHROETER (U.S.P. 1,582,310, 27.4.26. Appl., 29.8.21).—See G.P. 324,861; B., 1921, 253 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chlorination of wool. S. R. and E. R. TROTMAN (J.S.C.I., 1926, **45**, 111—115 T).—The authors have shown (J.S.C.I., 1922, 219 T) that wool is damaged more quickly by chlorine than by hypochlorous acid, and that the structural damage caused during chlorination is due to the presence of free chlorine in the chlorinating liquors. The destruction of the epithelial scales of wool, while diminishing the felting power, does not produce unshrinkability. There is no relation between damage and shrinkage, the former being merely a secondary effect. It is impossible to produce absolute unshrinkability by means of chlorine alone. Felting and shrinking appear to be distinct phenomena depending upon the different proteins present in the epithelium and cortex respectively (cf. J.S.C.I., 1926, 20 T). The action of chlorine on wool proceeds by both adsorption and chemical combination. When chlorine is used as hypochlorous acid, neither damage nor loss of weight is produced until over 4% of chlorine on the weight of the wool has been absorbed (cf. E.P. 239,360; B., 1925, 844). In the case of chlorine, serious damage is apparent, even when small percentages are employed and the rate of adsorption is affected by the presence of acid. It is possible that hypochlorous acid may act by liberation of nascent oxygen. Other oxidising agents investigated reduced felting, proportionately to the number of epithelial scales destroyed, but no marked decrease

in shrinkage was observed. The action of hypochlorous acid is fundamentally different from that of chlorine.

Influence of atmospheric humidity on the absorption of moisture by textile fibres. J. OBERMILLER and M. GOERTZ (Textilber., 1926, 7, 71—76).—The moisture absorption of various types of fat-extracted cotton, wool, natural (not fat-extracted) and artificial cellulose silks was determined after prolonged exposure at 20° to air, the relative humidity of which was successively maintained (within 2% limit of error) at 2·5%, 35%, 55%, 75%, 92%, and 97% by contact with potassium sulphate, crystalline sodium carbonate, potassium chloride, calcium nitrate, crystalline calcium chloride, and anhydrous calcium chloride respectively. Previous treatment considerably influences the absorption of moisture by all textile materials; *e.g.*, raw silk before drying and after drying for 72 and 177 hrs. at 75—100° absorbed 15%, 13·02%, and 12·33% of moisture when exposed afterwards at 20° to air of 75% relative humidity. Previous drying at a high temperature decreases the subsequent rate of absorption of moisture. The average moisture contents of cotton (American and Indian), wool (pure and chrome-mordanted), raw silk, degummed silk, and artificial (cuprammonium and viscose) silks exposed in air of 2·5%, 35%, 55%, 75%, 92%, and 97% relative humidity, and at 20° are 1·3, 5·5, 8·0, 11·0, 16·5, and 22·0%; 1·8, 11·0, 15·0, 18·5, 25·0, and 29·0%; 2·0, 8·0, 11·5, 15·5, 23·0, and 32·0%; 1·8, 7·3, 10·0, 13·5, 21·0, and 29·0%; 2·5, 8·5, 12·5, 17·5, 28·0, and 38·0 respectively. The higher moisture absorption of raw silk indicates that silk gum (sericin) is more hygroscopic than pure silk (fibroin). Indian cotton and viscose silk have slightly greater affinities for moisture than American cotton and cuprammonium silk respectively. The moisture regains of cotton, wool, raw silk, degummed silk, and artificial silks exposed at 20° in air of 65% relative humidity are 9·5%, 16·5—17·0%, 13·5%, 11·5—12·0%, and 15·0% respectively, as compared with the international standard regains of 8·5% (cotton), 18·25% (wool), and 11·0% (natural and artificial silks). The maximum moisture contents of cotton, wool, raw silk, degummed silk, and artificial silk exposed at 20° in air of 100% relative humidity are greater than 26%, 32%, 35%, 35%, and 40% respectively, the results of Scheurer (cf. B., 1921, 359 A) being thereby confirmed. The absorption of moisture by textile materials is considerably decreased by rise of temperature, but is not appreciably affected by changes of barometric pressure. Dry cotton, wool, raw and degummed silk, cuprammonium and viscose silks suffer a considerable rise in temperature during subsequent absorption of moisture. A. J. HALL.

Relationship between the [physical] structure of artificial silks and their resistance to attack by enzymes. P. KARRER (Textilber., 1926, 7, 23—24; cf. B., 1925, 441, 585).—Scoured cotton, the same cotton mercerised, pure filter paper, cuprammonium silk of 120 denier, and cellulose regenerated from cellulose triacetate suffer 2·1%, 10·2%, 7·1%, 30·8%, and 70·0% hydrolysis respectively, when exposed under similar conditions to the action of an enzyme extracted from the intestines of vineyard snails. Cuprammonium silk and other regenerated celluloses may be quantitatively converted into dextrose by means of snail enzymes, but raw cotton even by successive treatments with such enzymes cannot be hydrolysed more than 25%. Raw cotton apparently consists of two types of cellulose distinguished by their resistance to attack by enzymes. It is suggested that something other than structural chemical change is responsible for the increased susceptibility to attack by enzymes of regenerated or alkali-treated celluloses. "Milky" patches sometimes found in commercial viscose silk materials contain fibres which have abnormal increased susceptibility to attack by snail enzymes, but which have the same microscopic characters, the same moisture content and swelling properties as normal viscose fibres, and yield similar *X*-ray spectrograms. Differences in the behaviour of various types of cellulose towards snail enzymes correspond to differences in their dyeing and absorptive properties. A. J. HALL.

Thorne bleaching process [for cellulose]. J. FUNCKE (Papier-Fabr., 1926, 24, 217—220).—Cellulose pulp is concentrated to 7—9% in a drying cylinder, and then to 35% in a screw press. The material emerging from the press passes into a mixer, where the requisite amounts of bleach liquor and water are added, the volume and temperature of the water being adjusted so that the mixture contains 20% of cellulose at 20°. This then passes into a bleaching tower provided with an air-blast, and at the exit at the bottom it is diluted with water to a concentration of 1%, and so washed. Bleaching may be completed either by a repetition of this process or in a hollander. This continuous process, working at a high cellulose concentration, effects economies in chlorine (30—40%), steam (50%), and time (30%), and the product is stronger, has a higher cellulose content, and does not turn yellow on keeping. A. GEAKE.

Researches on viscose. III. Preparation of alkali-cellulose. G. KITA, R. TOMISHISA, and H. ICHIKAWA (Cellulose Ind., Tokyo, 1926, 2, 67—79; cf. B., 1926, 45).—When the alkali-cellulose was xanthated immediately after steeping and pressing, the optimum results were obtained with a period of immersion in the mercerising liquor of 24 hrs. at 8°, the proportion of combined alkali and the viscosity of the viscose being then at a maximum. The temperature and time of ageing of the alkali-cellulose have little influence on the proportion of combined alkali, but a very strong influence on the viscosity of the viscose even when the ageing takes place in an atmosphere of hydrogen. Viscose prepared from alkali-cellulose aged at a relatively high temperature (20°) undergoes only small changes in viscosity during ripening. The proportion of combined alkali in the viscose tends to be lower the higher the concentration of sodium hydroxide in the steeping liquor. An equilibrium tends to be established between the sodium hydroxide in the alkali-

cellulose and the carbon disulphide with which it is brought in contact, so that with an excessive quantity of alkali a larger proportion of the carbon disulphide reacts with the soda and less alkali is combined as xanthate unless sufficient carbon disulphide is present to cover both requirements. The maximum proportion of combined alkali, up to 0·5 mol. per mol. of cellulose, is attained if the reaction is performed at 0°, or if alkali-cellulose containing a high concentration of sodium hydroxide is treated with a large excess of carbon disulphide. A low temperature of xanthation together with a low temperature of ripening favours the maximum combination of alkali in the form of xanthate and its preservation during a prolonged period. J. F. BRIGGS.

Laboratory preparation of viscose. E. H. MORSE (Ind. Eng. Chem., 1926, **18**, 398—400).—To prepare on the laboratory scale viscose grades comparable with commercial grades, the commercial procedure must be modified. The alkali concentration for the first stage of treatment should be 20—21% at room temperature. Pressing may be carried out in a letter-press and crumbing by means of any sharp pointed instrument. The fluffy mass of crumbs must be kept for about 24 hrs. at a temperature between 18° and 23°, after which it is mixed in a revolving container with a quantity of carbon disulphide equal to rather more than a third of the weight of α-cellulose in the original pulp; the disulphide is added in four portions at room temperature. Ripening proceeds satisfactorily at room temperature. For relatively thick films 2 drops of viscose should dissolve clear in 10 c.c. of 2·5—3% of magnesium-free sodium chloride solution, but not in a stronger solution. Filtering through several layers of fine linen, or cotton wool between fine bleached muslin, should not be carried out until the solution has been kept for at least 24 hrs., and two filtrations are necessary if the solution is to be used to spin fine filaments. For general use a viscosity such that a steel ball 3 mm. diameter will fall 100 mm. through the solution in 15 sec. is satisfactory, and may be altered by addition of small amounts of clear 15% caustic soda solution. D. G. HEWER.

Apparent viscosity of solutions of nitro-cotton in various solvents. J. W. McBAIN, C. E. HARVEY, and L. E. SMITH (J. Physical Chem., 1926, **30**, 312—352; cf. Baker., J.C.S., 1913, **103**, 1653; Piest, B., 1911, 835; Mardles, J.S.C.I., 1923, **42**, 129, 211 T).—The apparent viscosity of solutions of nitro-cotton depends on the rate of shear and hence is not a true viscosity. It is so great that it must be ascribed to interference between ramifying aggregates of colloidal particles in the solution. No accepted formula represents the dependence of viscosity on concentration, the more concentrated solutions being less than proportionately viscous (cf. Hatschek, Biochem. J., 1916, **10**, 325; Baker, *loc. cit.*; Mardles, J.C.S., 1923, **123**, 1951). Solutions which have aged for considerable periods show viscosity more nearly proportional to concentration. The temperature coefficient is very large. The period of drying the cotton at 60° affects the viscosity of solutions made from it, the most viscous resulting from material which has been heated for less than 24 hrs.; when the heating is extended to weeks or months the viscosity is progressively less. Vigorous shaking of the solutions diminishes the viscosity at 55°, presumably by breaking down the residual structure derived from the original fibres. The greatest effect occurs at the beginning when the largest aggregates are being disintegrated, whilst at a later period the effect becomes negligible. The higher the temperature of preparation the lower is the viscosity; the difference between solutions prepared at 55° and 90° showed no change after several months.

The behaviour of all known mixtures of solvent is tabulated. In the majority of cases a mixture in suitable proportions is a better solvent than the constituents of the mixture alone, and the solvent power of a given substance is largely dependent on adventitious impurities; generally speaking, the purer the solvent the less its efficiency as such. Data for fifteen solvents show that at 55° the viscosity of one definite sample of nitro-cotton in these solvents is inversely proportional to their solvent power (cf. Duclaux and Wollman, B., 1920, 528 A); low relative viscosity is a general measure of solvent power, the best solvents giving the least viscous solutions; and to a limited extent, increase in mol. wt. of comparable compounds lowers solvent power. The formanilides appear to be a better group of solvents than the urethanes or the pure esters. Viscosity in any solvent diminishes on keeping at 55° or at 90°, and, with the possible exception of benzylformanilide, is approximately proportional to the logarithm of the time. The changes in viscosity are very large, but they are in accord with the hypothesis that the best solvents are those which most quickly and effectively resolve the colloidal aggregates into the smallest particles. The higher the temperature the quicker and the greater the amount of degradation, but no definite end value is reached. Nitration analyses show that the ageing cannot be due to the denitration of the nitro-cotton by the solvent. Nitro-cotton recovered by precipitation with water or by evaporation from solution in acetone is much more difficult to dissolve than the original nitro-cotton, and the resulting viscosity may be affected and even increased. Anhydrous alcohol is a complete solvent for propulsive soluble nitro-cotton at the temperature of a carbon dioxide–ether mixture, whilst, at the ordinary temperature, the sol sets to a stable jelly, which shows the reverse change on cooling even after a period of 18 months. Syneresis is also shown to a marked degree. The imperfect solution obtained with alcohol at 130° is due to impurities resulting from denitration and decomposition. A theory of the behaviour of solvents towards nitro-cotton, which should be applicable throughout colloid chemistry, is advanced. L. S. THEOBALD.

See also A., May, 468, **Formation of an alkali-cellulose compound in an aqueous alcoholic medium** (KATZ). 502, **Depolymerisation of cellulose** (HEUSER and HIEMER). 547, **Incrustations**

of flax (Ehrlich and Schubert). 548, Incrustive substances of plants (Heuser) ; Leaf-tissue fluids of Egyptian cottons (Harris and others).

Patents.

Reducing the inflammability of nitrocellulose threads. J. R. Lavaud (F.P. 600,852, 17.7.25. Conv., 23.4.25).—The material is successively treated with 10% acetic acid or 5% formic acid for 30—40 min., centrifuged, treated with a 10% solution of crystalline sodium sulphide and crystalline magnesium sulphate for $2\frac{1}{2}$—3 hrs. at 25—30°, steeped in 5% sodium hyposulphite solution, dipped in a 10% solution of crystalline aluminium chloride, and treated for 30 min. in a bath containing 8 pts. of ammonium sulphate, $2\frac{1}{2}$ pts. of commercial ammonium carbonate, 3 pts. of boric acid, and $1\frac{1}{2}$ pts. of borax, after which it is rinsed and dried.
L. A. Coles.

Producing acid liquor in sulphite pulp processes. J. D. Jenssen, Assr. to G. D. Jenssen Co. (U.S.P. 1,571,271, 2.2.26. Appl., 24.2.25).—The plant comprises a primary (weak liquor) tower for the preliminary step, a secondary (strong liquor) tower for further treatment, and a mixer for heating the liquor in its passage from the primary to the secondary tower. J. F. Briggs.

Process of hydration in paper-making, using friction. J. A. De Cew (U.S.P. 1,578,405, 30.3.26. Appl., 29.1.25).—The cellulose is beaten and "jordaned" (refined) in water containing cold dilute caustic alkali and the fibres are then sized by a special series of operations in which the alkalinity is neutralised. J. F. Briggs.

Acetylation of cellulose. Soc. Chim. des Usines du Rhône (E.P. 231,837, 24.1.25. Conv., 2.4.24).—See U.S.P. 1,566,398 ; B., 1926, 152.

Plastic mass [from cellulose ethers]. L. Lilienfeld (U.S.P. 1,563,204, 24.11.25. Appl., 11.1.21).—See E.P. 149,319 ; B., 1922, 95 a.

Manufacture of artificial silk and like materials from viscose solutions. W. P. Dreaper (U.S.P. 1,583,534, 4.5.26. Appl., 18.10.23). —See E.P. 215,028 ; B., 1924, 592.

Preparation of threads from cellulose acetate. Farbenfabr. vorm. F. Bayer & Co., Assees. of E. Hubert, O. Leuchs, and L. Lock (U.S.P. 1,583,717, 4.5.26. Appl., 14.7.24).—See E.P. 219,333 ; B., 1925, 843.

Asbestos paper etc. N. Sulzberger (U.S.P. 1,581,618–9, 20.4.26. Appl., 30.4 and 18.8.21).—See E.P. 186,409 ; B., 1922, 894 a.

Treatment of wood (F.P. 586,856).—See II.

Separating alkali hydroxides from solutions (E.P. 246,355).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching artificial silk with "Aktivin" [sodium p-toluenesulphochloramide]. R. Feibelmann (Textilber., 1926, 7, 47—48).—Aktivin is a satisfactory substitute for hypochlorites and the like in the bleaching of viscose silk. Raw silk is bleached to a uniform pale cream shade without deterioration by treatment with a 3—4% solution of Aktivin at 60—70° ; the resulting loss of activity of the Aktivin is strictly proportional to the amount of bleaching effected, and the residual liquor may be used again after titration with potassium iodide and sodium thiosulphate and addition of a suitable amount of Aktivin. White is obtained by bleaching with a 0·3% solution of Aktivin containing 0·5% of 50% acetic or formic acid. Under these conditions the bleaching solution is slightly cloudy owing to the separation of *p*-toluenesulphochloramide, but no deterioration of the bleached silk occurs even when high temperatures are used. Viscose silk materials bleached by means of Aktivin have a more uniform affinity for dyes than those bleached by other methods. A. J. Hall.

Determining the degree of mercerisation of cotton fabric. R. Haller (Textilber., 1926, 7, 65—66).—Measurement of the amounts of white, black, and pure colour tone in dyed mercerised cotton fabric by means of the Ostwald method is used for the determination of the degree of mercerisation of the dyed fabric, the method being rapid but somewhat less accurate than the titanous chloride method of Knecht (B., 1905, 154). Examination of the colour contents of cotton fabrics mercerised with caustic soda of 2—30° B. (*d* 1·014—1·261) and afterwards dyed with Diamine Blue 3R, Benzo Azurine, and Congo Red showed that the black and white contents of the resulting shades remained constant from 2 to 12° B. (*d* 1·014—1·091), then distinctly increased and decreased respectively from 12° to 26° B. (*d* 1·091—1·221), and then remained constant ; simultaneously the content of pure colour decreased similarly but less regularly. It is concluded that the chief mercerisation effects are produced by caustic soda of 12° to 26° B., although Knecht (B., 1908, 400) found that mercerisation distinctly commences with caustic soda of 7° B. (*d* 1·052). White cotton suffers a slight darkening during mercerisation. A. J. Hall.

Physics and chemistry of dyeing processes. II. Dyeing of non-denitrated nitrocellulose silk. K. H. Meyer, C. Schuster, and W. Bülow (Textilber., 1926, 7, 29—31 ; cf. B., 1926, 316).—Nitrocellulose silk absorbs *o*-nitroaniline and benzamide from aqueous solutions in accordance with Henry's law, subject to correction in the case of benzamide for the molecular association which it suffers in concentrated aqueous solution. The solubilities of *o*-nitroaniline and benzamide in nitrocellulose silk are 250 and 30 times greater respectively than their corresponding solubilities in water. One gram of nitrocellulose silk is capable of absorbing (dissolving) a maximum of 1·25 g. of *o*-nitroaniline but absorbs only 0·9 g. when immersed for $1\frac{1}{2}$ months in an aqueous solution maintained saturated with *o*-nitroaniline. Nitrocellulose silk behaves similarly to cellulose acetate silk in respect

of its absorption of *o*-nitroaniline from aqueous solution and its affinity for feebly basic and non-sulphonated dyes. Cotton cellulose nitrated so that it contains 12% N behaves similarly to nitro-cellulose silk. A. J. Hall.

[Dyeing] cellulose acetate silk effects [in union fabrics]. G. Rudolph (Kunstseide, 1926, **8**, 13—15).—Blue, red, and white effects in cotton–wool–cellulose acetate silk union fabric are obtained by dyeing the wool with a (blue) acid dye from an acid bath, the cotton with a (red) cotton dye at 50° in the presence of 3% of Katanol W (for retarding absorption of the cotton dye by the wool), and leaving the cellulose acetate silk white. Three-colour effects on similar fabric are obtained by first dyeing the cellulose acetate silk with Cellit Fast Yellow 2GN and developing a red shade by the usual processes of diazotisation and coupling (the wool being thereby stained yellow and the cotton not affected), and afterwards dyeing the wool with a (blue) acid dye, the fabric being then thoroughly washed and the cotton dyed green or olive with a direct dye in the presence of Katanol W. Four colours are obtained on cotton–wool–natural and cellulose acetate silk union fabric by first dyeing the cellulose acetate silk red as described above, then dyeing the wool and silk together with wool dyes having different affinities for wool and silk under different conditions, *e.g.*, Patent Blue and Tartrazine, *e.g.*, by dyeing for ½–¾ hr. at a low temperature (the silk thereby becomes dyed to a deep peacock blue) and afterwards adding sulphuric acid to the dye liquor and heating it to about 100° (the wool thereby becomes dyed Russian Green); subsequently the cotton is dyed with a violet direct dye in the presence of Katanol W at 50°. Three colours are obtained on cotton–viscose and cellulose acetate silk or cotton–mercerised cotton–cellulose acetate silk fabrics by previously dyeing the cotton (black) when in the form of yarn and afterwards dyeing the cellulose acetate silk and mercerised cotton or viscose silk in different colours by the usual methods. A. J. Hall.

[Use of] Katanol W [in dyeing union fabrics]. G. Rudolph (Dtsch. Färber-Ztg., 1925, 107. Textilber., 1926, **7**, 88).—The presence of Katanol W (sulphurised phenol) in dye-baths used for dyeing half-silk and half-wool materials considerably retards absorption by the animal fibres of direct cotton dyes, the dyeing of the cotton being unaffected. This restraining influence of Katanol W is used to facilitate the production of solid shades on half-wool fabrics by the neutral single-bath process of dyeing; the wool is dyed to shade first at a high temperature, the dye-bath is then cooled to 75—80°, 3% of Katanol W added, and the cotton allowed to absorb colour until the desired shade is obtained, there being no simultaneous change of shade of the wool. Katanol W is not added at the commencement of the dyeing process since it also retards the absorption by wool of certain acid dyes which have an affinity for wool in a neutral bath. Cotton in half wool fabric containing wool dyed with an acid dye is preferably dyed with substantive dyes at 75—80° in the presence of Katanol W since the dyed cotton is faster to rubbing, the wool is less stained and deteriorated in handle, and the dye-bath more exhausted than in the usual method of dyeing at 30° in the presence of sodium carbonate. Katanol W may be of assistance in the dyeing of half silk materials, but natural silk better resists direct dyes when pre-mordanted for 1 hr. at 80—90° with 8—10% of Katanol W and 3—4% of formic acid. A. J. Hall.

[Dyeing] Naphthol AS colours on artificial silks. H. Lint (Textilber., 1926, **7**, 46).—A satisfactory range of colours very fast to all influences are obtained on artificial silks by means of Naphthol AS dyes. In dyeing, the silk is treated for 20—30 min. at 25—30° with a solution of a suitable Naphthol AS compound, centrifuged without previous rinsing, and then treated for 15—20 min. with a cold solution containing a diazotised amine. The dyed silk is thoroughly washed, soaped for 20—30 min. at 60—80° in a solution containing 4—5 g. of soap per litre, again thoroughly washed, and then dried after brightening the shade by treatment in dilute acetic or formic acid. Naphthol AS colours dyed on crude viscose silk are not changed in shade by the usual subsequent process of desulphurisation. A. J. Hall.

Use of Aktivin for finishing printed fabrics and window-curtain materials. R. Feibelmann (Textilber., 1926, **7**, 144—146).—Neutral, transparent, homogeneous, highly glutinous starch pastes containing no dextrin, maltose, glucose, or other sugar substances and suitable for finishing textile fabrics are prepared by boiling aqueous suspensions of starch (maize, rice, wheat, potato, etc.) containing about 1% (calc. on the weight of starch) of Aktivin (sodium *p*-toluenesulphochloramide); the viscosity of the resulting pastes is lowered by prolonged boiling, but no formation of reducing sugars occurs. This method of preparing finishing pastes is superior to processes now used in which starch is subjected to the action of diastatic enzymes, since the conditions of preparation (concentration of Aktivin and time) may be varied widely without the danger of formation of dextrin and lower degradation products of starch which ultimately promote the yellowing of white fabrics during storage. Starch pastes prepared with Aktivin are transparent only when hot, for insoluble starch separates when they are cooled; transparency is regained when the paste is reheated. A paste suitable for finishing white (lace) curtain materials is prepared by boiling a mixture of 10 kg. of potato starch, 250—270 litres of water, and 100 g. of Aktivin for 10—15 min., and then adding 1—2 kg. of Turkey-red oil, soap, or other suitable fatty substance. A paste for finishing printed fabrics is prepared by boiling a mixture containing 10 kg. of potato starch, 80 litres of water, 100 g. of Aktivin, and 20 g. of sodium carbonate for 15 min., afterwards adding 4 litres of 10% solution of soap and 1 litre of Turkey-red oil; suitable weighting substances or hygroscopic compounds (*e.g.*, glycerin) for conferring

a linen-like handle to the finished fabric may be added. A. J. HALL.

PATENTS.

Weighting fibres. O. BERG and M. IMHOFF (U.S.P. 1,579,628, 6.4.26. Appl., 26.12.22).—Textile material is weighted with tin by any method including the use of a fixing bath, and is subsequently treated with a soluble lead compound. L. A. COLES.

Production of pattern effects on woven fabrics. HEBERLEIN & Co. A.-G. (E.P. 237,909, 28.7.25. Conv., 29.7.24).—Textile material containing cellulose acetate silk interwoven with, *e.g.*, cotton threads, is printed with a reserve, and subsequently treated with a solvent, such as chloroform, acetone, pyridine, or epichlorohydrin, capable of dissolving out the artificial silk not protected by the reserve. The reserve may protect the artificial silk mechanically by forming a layer insoluble in the solvent, glue solution and alcoholic shellac solution being suitable for the purpose, or it may contain alkalis, which hydrolyse the cellulose acetate and thereby render it insoluble in the solvents. Various modifications are obtained by introducing dyeing or printing processes to obtain different colour effects. L. A. COLES.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Nitrogen fixation in the high-tension arc. H. V. TARTAR and M. F. PERKINS (J. Physical Chem., 1926, **30**, 595—619).—The effect of pressure on the yield of fixed nitrogen, the equilibria obtained with various mixtures of nitrogen and oxygen, and the effect of moisture on the yield when air is used have been examined in a laboratory arc furnace which can be made to give an efficiency almost equal to that obtained in commercial practice. Under constant conditions, the concentration of nitric oxide for any given mixture between the limits oxygen 1 vol., nitrogen 4 vols., and oxygen 4 vols., and nitrogen 1 vol., decreased linearly with increasing velocity, but the yield of fixed nitrogen reached a maximum at an intermediate velocity, which is dependent to some extent on the composition of the gas and its rate of flow. Very high air velocities gave a low output in respect to both yield and concentration of fixed nitrogen. A gradual decrease in concentration of nitric oxide and in the yield per kw.-hr. accompanies a reduced working pressure, and at corresponding pressures a mixture of equal volumes of nitrogen and oxygen gave the greatest yield. No marked improvement in yield by using decreased pressure could be detected. Experiments with various mixtures of nitrogen and oxygen using different amperages showed that the mass action law holds good for the equilibria obtained. Cooling from the whirling arc was very effective. A method is given for the calculation of the concentration of nitric oxide resulting from repeated exposures of a nitrogen–oxygen mixture to a high-tension arc. The presence of water-vapour in the air supplied to the arc reduced both the yield and the concentration of the nitric oxide. With 2·6% of moisture the yield was reduced by 25%. L. S. THEOBALD.

High-test bleaching powder—calcium hypochlorite and basic calcium hypochlorite. S. URANO (Trans. Amer. Electrochem. Soc., 1926, **49**, 303—311. Advance copy).—Basic calcium hypochlorite, $Ca(OCl)_2,2Ca(OH)_2$, is obtained by adding pure slaked lime to a clear solution of bleaching powder (5—6% of available chlorine) and evaporating *in vacuo* at 40°. In definite conditions of concentration of the liquid phase the basic salt crystallises and may be separated, washed, and dried *in vacuo* at 80°. In this way a bleaching powder containing 40—48% of available chlorine is obtained. The salt is stable at above 115°, odourless, and not deliquescent. The rate of loss of chlorine, whether the salt is wet or dry, is less than in the case of ordinary bleaching powder. On adding water to the basic salt the calcium hypochlorite passes into solution and if the calcium hydroxide is filtered off, this solution may be concentrated *in vacuo* until normal calcium hypochlorite (90—95% of available chlorine) crystallises. This material is also relatively stable at 40—50°. Above 150° it is decomposed into calcium chloride and oxygen with violent explosions. C. IRWIN.

Pyro- and hydro-treatment of magnesite and dolomite. H. M. HENTON (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy, 34 pp.).—Experiments on the separation of magnesia from dolomite were conducted on a laboratory scale and later on in an extended plant situated near the quarries and utilising carbon dioxide from the calcining kilns. When calcined no carbon dioxide was evolved from dolomite at any temperature near 450°, the dissociation point of pure magnesium carbonate, and it seems evident that energy is required to break up a natural combination of the calcium and magnesium carbonates. At 500° magnesium carbonate was decomposed to the extent of 50% and complete decomposition was effected in $1\frac{1}{2}$ hrs. at 600° under a very slight partial pressure of carbon dioxide. This stage of calcination, in which the calcium carbonate remains undecomposed, is the most suitable for the after-extraction process and may be effected in a rotary kiln. No advantage in calcining was obtained by reducing the size of the material below $\frac{1}{4}$ in. Carbon dioxide driven off during calcination was cooled, cleaned, and compressed for use in converting the magnesium oxide into the soluble bicarbonate. The apparatus used for carbonating a slurry of the calcined material was of the "Pachuca" type in which a vertical cylindrical vessel having a conical bottom contains a central tube which carries the liberated compressed gas and rising slurry on the same principle as the air lift. The best results were obtained by carbonating at atmospheric pressure with a gas mixture of the composition CO_2 25%, air 75%, and a number of tests showed from 87·3 to 96·4% extraction of magnesium oxide, using four stages of extraction. A

nearly clear solution of magnesium bicarbonate may be decanted, filtered, and then precipitated by heating, and although complete precipitation was effected usually below 100°, boiling seemed necessary in order to cause a quick settling of the precipitate. Long needles were observed frequently at the edges of a supersaturated solution of the bicarbonate before boiling though magnesium bicarbonate is known only in solution. C. A. KING.

Transportation of liquefied chlorine gas. H. P. WELLS, H. M. MABEY, and J. M. ROWLAND (Trans. Amer. Electrochem. Soc., 1926, 49, 269—284. Advance copy).—Liquid chlorine is distributed in America by means of cylinders of 10—150 lb. capacity, in 13-ton tank wagons, and in multiple-unit tank wagons comprising 15 1-ton containers loaded on a suitable underframe and removable as required. Similar containers are suitable for sea transport. In all cases specifications and testing are controlled by the Inter-State Commerce Commission. The cylinders are tested to 1000 lb. hydrostatic pressure when new and to 500 lb. after five years. They are fitted with safety plugs of fusible alloy, as are also the 1-ton containers. These, which are tested to 500 lb. per sq. in., have concave ends for convenience in clamping and lifting. Both types of container are filled by weight. Tank wagons are fitted with top discharge only and are insulated with pressed cork lining covered by steel plate. Valve parts in contact with the gas are of monel metal. Chlorine can be withdrawn as either gas or liquid through different valves. If necessary air pressure or a steam coil may be used to assist discharge. Vapour pressure curves of chlorine and of chlorine plus air are given. C. IRWIN.

See also A., May, 466, **Dithionic acid and its salts** (DE BAAT). 474, **Thermal dissociation of sodium cyanide** (GUERNSEY and SHERMAN). 475, **System water–aluminium oxide** (GUICHARD). 482, **Metals and nitric acid** (KLEMENC). 487, **Action of silica on metal sulphates** (MARCHAL); **Oxidation of ammonia** (PARTINGTON); **Action of hydrogen and water on phosphorus at high temperature under pressure** (IPATIEV and NIKOLAJEV). 489, **Preparation of cyanogen** (RICCA); **Preservation of gaseous cyanogen** (RICCA); **Determination of perchloric acid** (LOEBICH); **Hydrazine sulphate as standardising reagent for iodine solutions** (CATTELAIN). 490, **Electrometric titration of oxidising acids** (MALAPRADE); **Determination of nitrogen, nitrate, chlorate, and perchlorate** (KÜRSCHNER and SCHARRER); **Colorimetric determination of hydroxylamine** (PUCHER and DAY); **Determination of phosphorous and hypophosphorous acids and calcium hypophosphite** (ZIVY); **Determination of arsenic acid** (FRANCIS).

Preparation of industrial sands. WEIGEL.—See I.

Insoluble anodes for electrolysis of brine. FINK and PAN.—See XI.

PATENTS.

Manufacturing sulphuric acid. H. PETERSEN (E.P. 249,914, 29.12.24).—In the chamber process, an intimate mixture of gases and liquid is obtained by using two grades of finely divided filling material not exceeding 40 mm. size on the average. The finer material of 1—10 mm. size is used in the parts of the plant where nitrogen oxides are absorbed, whilst pieces the size of a walnut are preferably used in the denitration towers. The filling material is contained in chambers of greater width than height, the gases being forced in a vertical direction either upwards or downwards by means of a fan. One or more additional units may be placed between the denitrating and absorbing parts of the plant and rinsed with water or weak sulphuric acid, the liquid circulating in these units not being allowed to enter the other parts of the system, whilst the gases flow in a downward direction and thus become thoroughly mixed and distributed over the entire sectional area of the units. H. ROYAL-DAWSON.

Recovering or separating caustic [alkali] hydroxides from solutions containing them. J. Y. JOHNSON. From VISCOSE Co. (E.P. 246,355, 23.10.24).—The alkali hydroxide contained in waste liquors resulting from mercerisation, the digestion of pulp, and similar operations is recovered by dialysis. The process is preferably worked on the counter-current principle, using a number of dialysing cells in series. Woven fabric, *e.g.*, cotton sheeting, parchmentised by immersion for 2 min. in sulphuric acid, *d* 1·10, at a temperature below 30°, may be conveniently used as the membrane. D. J. NORMAN.

Manufacturing hydrocarbons and cyanides. J. C. CLANCY (U.S.P. 1,567,241, 29.12.25. Appl., 18.4.25).—Comminuted carbonaceous material is destructively distilled in contact with molten alkali cyanide. The volatile products of the reaction, largely hydrocarbon oils, are collected and the residue is subjected to a cyanide-producing reaction, *e.g.*, by mixing it with sodium carbonate and treating at a high temperature with nitrogen. C. IRWIN.

Method of making calcium cyanide. F. J. METZGER, Assr. to CALIFORNIA CYANIDE Co., INC. (U.S.P. 1,573,732, 16.2.26. Appl., 23.11.25; cf. B., 1926, 272).—Finely divided calcium carbide (100 pts.) is added to a mixture of 400 pts. of hydrocyanic acid containing about 1% of water; if lime be present in the carbide sufficient water is formed in the reaction. The mixture is maintained at 60° for about 2 hrs. The product is calcium cyanide of the formula $Ca(CN)_2,xHCN$, where x is approximately 2; it is comparatively safe to handle and when exposed to air gives off hydrocyanic acid so that it is of value as an insecticide and the like. T. S. WHEELER.

Process for making (A) heavy basic carbonate of magnesium, and (B) light basic carbonate of magnesium. B. B. GRUNWALD (U.S.P. 1,573,603–4, 16.2.26. Appl., 30.6.24).—(A) Calcined magnesite

is mixed with 10—15 pts. of water and treated with carbon dioxide so that when carbonation is finished the temperature has reached 38°. The product is much denser than basic magnesium carbonate as usually prepared and does not shrink on drying. It is of value as a cement and binder in the preparation of heat-insulating materials (cf. B), and as a filler for paper pulp and the like. The product obtained by mixing with asbestos and calcining at about 100° has great strength and good heat-insulating properties. (B) Heavy basic magnesium carbonate prepared as in (A) is agitated and heated with water to about 45° and kept for 15 hrs. when expansion is complete. Alternatively the heavy basic carbonate is heated to 60° with water until sufficient expansion has occurred, when cold water is added to stop further expansion. The product is very light, does not shrink in drying, and is of value as a heat-insulating medium. For storage purposes the light basic carbonate is prepared by heating the heavy material with water at 80° until expansion is complete. When required it is mixed with a suitable quantity of heavy basic magnesium carbonate, as when completely expanded at 80° it is too light to be of value. T. S. Wheeler.

Silicious alkaline-earth product. R. Calvert, Assr. to Celite Co. (U.S.P. 1,574,363, 23.2.26. Appl., 1.12.23).—Diatomaceous earth is heated with excess of water and 5—80% of lime or other alkaline-earth hydroxide at about 100° until all expansion ceases. The product is very finely divided and is hence more efficient than diatomaceous earth as a filter aid, and a decolorising agent. T. S. Wheeler.

Production of potassium sulphite, bisulphite, and metabisulphite. E. Hene (G.P. 424,949, 2.3.24).—Potassium bisulphite solution containing a relatively high proportion of calcium ions, is prepared by treating calcium bisulphite solution, with or without the addition of solid calcium sulphite, with potassium sulphate, solid or in solution, or with syngenite. After filtration, the solution is treated with potassium sulphate so as to obtain a solution containing relatively few calcium ions, and the precipitate, which contains a high percentage of potassium, is used for treating further quantities of calcium bisulphite solution. The process may be carried out at low temperatures, or under increased pressure. Calcium sulphite used in the process is obtained by heating calcium bisulphite solution to expel sulphur dioxide. The potassium bisulphite solution obtained is purified by heating it, and filtering off precipitated calcium hydroxide and iron compounds. L. A. Coles.

Manufacture of nitrites from nitrous gases. A. Bähr (G.P. 424,950, 2.5.25).—Waste nitrous gases are dried and diluted with dry air free from carbon dioxide, before passing into absorption apparatus. If necessary, the nitrous gases are subjected to a reduction process before dilution with air. L. A. Coles.

Continuous production of anhydrous sodium sulphate. W. Recha (G.P. 424,983, 4.10.24).—Crystalline sodium sulphate, $Na_2SO_4,10H_2O$, is heated to a temperature above 32·4°, *e.g.*, in the trough of a rotating filter, and the anhydrous sodium sulphate which crystallises out is removed continuously from the liquor. L. A. Coles.

Stabilising ammonium carbonate, bicarbonate, and carbamate. A. and L. Welter, and A. Weindel (G.P. 425,335, 3.12.24).—Dry, finely powdered ammonium compounds, alone or mixed with organic or inorganic salts, and atomised inorganic salt solutions, or cellulose waste liquor, or mixtures of these, are charged simultaneously into the top of a tower in which the material is dried by contact with a counter-current of air, preferably charged with acid gases, *e.g.*, hydrogen chloride (cf. G.P. 336,612; B., 1921, 613 A). L. A. Coles.

Continuous purification of salts. I.-G. Farbenind. A.-G. (G.P. 425,503, 1.7.24).—The salt to be purified trickles down a fall tube through an ascending current of a saturated solution of pure salt of the same composition. Purified salt after deposition, and if necessary after dissolving in water added for that purpose, is withdrawn, whilst a further quantity of impure salt is fed to the fall tube, *e.g.*, by means of the wash liquor used in the purification. J. S. G. Thomas.

Production of potash and alumina from leucite. S. I. P. Soc. Ital. Potassa (G.P. 425,797, 24.1.24).—Leucite is heated with lime until the mixture fuses, and the product, in the form of big lumps, is allowed to cool slowly whereby it falls to a powder. L. A. Coles.

Manufacture of silica gel. Chem. Fabrik auf Actien (vorm. E. Schering), and W. Klaphake (E.P. 250,078, 20.7.25).—Silica gel previously deprived of the major part of its water content, or dried at ordinary temperature for several days, is heated for a short time to about 650° in a current of air. This has the advantage of causing any coloration of the gel to disappear, giving the product a more glass-like appearance. H. Royal-Dawson.

Manufacture of iron carbonyl. J. Y. Johnson. From Badische Anilin- & Soda-Fabr. (E.P. 250,132, 25.11.25).—In the manufacture of iron carbonyl by passing carbon monoxide over metallic iron at a high pressure, the speed of reaction is increased when the temperature is raised above 140°, and under such conditions the yield of product is considerably increased by reducing the temperature of the gases before releasing the pressure. The temperature of the reaction gases is preferably reduced by utilising the heat thereof, while the gases are still under high pressure, for preheating the fresh gases introduced into the apparatus. H. Royal-Dawson.

Manufacture of hydrogen. W. P. Rogers (E.P. 249,925, 1.1.25).—In the process in which iron oxide is reduced by water-gas and re-oxidised with

steam, smaller depths of the oxide (about 5 ft.) are used in the retorts, which are so arranged that the heating flues are heated by gas burnt in a central flue. While the oxide is reduced in one set of retorts, steam is reduced with formation of hydrogen in the adjoining set. The spent (oxidised) reducing gases from the retorts are returned to the water-gas generator where they are reduced by incandescent carbon and, after purification from hydrogen sulphide, are used again in the process. The generator is placed as near as possible to the retorts and the water-gas prior to entering the retorts passes through a heat-exchanger where it is heated by the hot gases from the oxidation retorts. H. ROYAL-DAWSON.

Recovery of sulphur from gases. F. RIEDEL (G.P. 425,664, 24.8.24).—Gases containing sulphur compounds, alone or mixed with air, are used in place of air in coal-dust or oil furnaces, contact material being added to the fuel, if necessary. Coal-dust is carried into the furnace by a blast of gas containing little or no oxygen, *e.g.*, flue gases or nitrogen. L. A. COLES.

Effecting catalytic reactions, particularly in catalytically producing hydrocyanic acid. BADISCHE ANILIN- & SODA-FABR., Assees. of A. MITTASCH and W. MICHAEL (U.S.P. 1,582,851, 27.4.26. Appl., 12.7.23).—See E.P. 220,771 ; B., 1924, 869.

Producing anhydrous magnesium chloride. C. ARNOLD. From DOW CHEMICAL CO. (E.P. 243,978, 27.4.25).—See U.S.P. 1,557,660 ; B., 1925, 991.

Purification or extraction of soluble substances [sodium nitrate from caliche]. W. BROADBRIDGE and W. G. SELLERS, Assrs. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,562,863, 24.11.25. Appl., 13.6.22).—See E.P. 182,859 ; B., 1922, 669 A.

Synthesis of ammonia. G. CLAUDE, Assr. to LAZOTE, INC. (U.S.P. 1,579,647, 6.4.26. Appl., 5.4.21).—See E.P. 161,195 ; B., 1922, 590 A.

Process of forming sulphite of lead. S. C. SMITH (U.S.P. 1,581,030, 13.4.26. Appl., 3.9.25).—See E.P. 239,558 ; B., 1925, 881.

Process of making lead sulphate. S. C. SMITH (U.S.P. 1,581,031, 13.4.26. Appl., 12.10.25).—See E.P. 239,559 ; B., 1925, 881.

Producing calcium hypochlorite compounds. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of G. PISTOR, H. S. SCHULTZE, and H. REITZ (U.S.P. 1,583,421, 4.5.26. Appl. 4.11.22).—See E.P. 188,662 ; B., 1923, 656 A.

Sodium compound. H. E. COCKSEDGE, Assr. to SOLVAY PROCESS CO. (U.S.P. 1,583,660, 4.5.26. Appl., 30.10.22).—See E.P. 202,678 ; B., 1923, 1020 A.

Process of forming sodium compounds. H. E. COCKSEDGE, Assr. to SOLVAY PROCESS CO. (U.S.P. 1,583,661, 4.5.26. Appl., 9.11.23).—See E.P. 206,372 ; B., 1924, 16.

Process of forming sodium carbonate. C. SUNDSTROM and G. N. TERZIEV, Assrs. to SOLVAY PROCESS CO. (U.S.P. 1,583,663, 4.5.26. Appl., 19.11.23).—See E.P. 223,015 ; B., 1924, 980.

High-pressure joints (E.P. 249,001).—See I.

Carbon monoxide from industrial gases (G.P. 425,418).—See II.

VIII.—GLASS; CERAMICS.

Chemical composition of window glass. E. P. ARTHUR (J. Amer. Ceram. Soc., 1926, **9**, 203—205).—Typical analyses of sheet glass made by various processes show variations (after allowing for minor constituents) of SiO_2 71·9 to 74·3%, CaO+MgO 11·1 to 14·2%, Na_2O 12·7 to 15·1%. Although the variations are small, the different glasses are distinct in properties, for the glass-making machines are sensitive to composition changes. The results of Morey and Bowen for the system SiO_2–CaO–Na_2O (B., 1925, 921) are confirmed in window-glass practice. For example, the devitrification temperature of the glass SiO_2 74%, CaO 11%, Na_2O 15% is given as about 1000°, whilst in practice, although the glass will work well at 993° a variation of 10° causes difficulty. Similar confirmation is obtained from hand-working conditions. A. COUSEN.

Deflocculation of clay slips and related properties. E. W. SCRIPTURE, JUN., and E. SCHRAMM (J. Amer. Ceram. Soc., 1926, **9**, 175—184).—Further experiments on a series of English china clays, ball clay, and kaolins (cf. B., 1925, 500) showed that an increase of the content of organic matter (as determined from the carbon content) coincided with an increase of toughness and plasticity. The change in the state of dispersion with variation of p_H for 20% slips was determined and proved the more plastic clays to have high dispersion with low p_H. The absorption of alkali in 20% and 40% slips was greater in the case of the more plastic clays. There appeared to be some relationship between p_H at the isoelectric point, the carbon content of the clay, absorptive power for caustic soda, and plasticity, although this could not be expressed in terms of any one variable. The viscosity–p_H curves for 40% slips were of the same general shape and location as the turbidity and settling curves. Casting slips were prepared in which the china clays and two ball clays were used in varying proportions. Ball clay was found to produce fluidity, and more viscous slips were obtained from more plastic than from less plastic china clays. A. COUSEN.

De-airing as corrective in drying [clay ware]. F. BRAND (J. Amer. Ceram. Soc., 1926, **9**, 188—190).—An examination of current views on the improvement in drying qualities found in the case of de-aired clays. Facts do not support the theory that the drying quality resides within the clay itself. A. COUSEN.

Heat required to fire ceramic bodies. A. E. MACGEE (J. Amer. Ceram. Soc., 1926, **9**, 206—247).

—Differential specific heats of ceramic materials, as determined by comparison with quartz, are firebrick about 0·43 (25—1200°), kaolin 0·5 (0—1000°), ball clay and diaspore clay 0·45 (0—1000°), flint and felspar 0·28 (0—1050°). Endothermic reactions occur with diaspore clay at 525°, firebrick and ball clay 550°, kaolin 575°, and exothermic processes with firebrick 950°, kaolin 960°, and ball clay (slight) 975°. The efficiencies of kilns tested were about 35%. A. Cousen.

Properties of silica brick from coke-oven walls. S. S. Cole (J. Amer. Ceram. Soc., 1926, 9, 197—202).—Silica brick samples made from different quartzites showed like properties after long service. They were in good condition after 12 years at operating temperatures of 1370—1430°, and the only vitrification was a glazed surface on the flue face. Little change had taken place in chemical composition or porosity. The quartz had been inverted, on the flue face chiefly to tridymite, on the coke face chiefly to cristobalite. A. Cousen.

Use of overglazes for polychrome terra-cotta. A. L. Bennett (J. Amer. Ceram. Soc., 1926, 9, 185—188).—Of the methods of polychroming terra-cotta, the use of overglazes to produce various colour effects in a single firing is economical and flexible. The overglaze should be insoluble in the liquid used and contain little or no clayey material. One difficulty lies in the tendency towards translucency and the influence of the character of the underlying glaze upon the colour reactions. A suggested base white overglaze is 0·621 ZnO, 0·379 CaO ; 0·453 Al_2O_3 ; 1·324 SiO_2, 0·126 SnO_2, which may be varied in shades of colour by blending the proper stains or colouring oxides. It is made up entirely from calcined or non-plastic materials. The methods of preparation and application of the glaze are described. A. Cousen.

Preparation of industrial sands. Weigel.—See I.

Treatment of magnesite and dolomite. Henton.—See VII.

Patents.

Making elongated articles of vitreous silica. Brit. Thomson-Houston Co., Assees. of B. F. Niedergesass (E.P. 241,544, 8.10.25. Conv., 20.10.24).—Particularly clear, bubble-free, and uniform elongated articles (as rods and tubes) of vitreous silica are produced by drawing directly from slugs of the vacuum-fused material, without contact with a die or solid body. A machine for performing this operation is described. A. Cousen.

Ovens or kilns [for ceramic ware etc.]. J. Laurent (E.P. 233,313, 24.2.25. Conv., 30.4.24). —The oven consists of a circular brickwork heating chamber with refractory lining. Around the periphery of the chamber is a number of fireboxes, each surrounded by a duct, opening to the atmosphere at one side and leading, on the other, to a chamber lying in the upper oven wall, between layers of refractory brick. Air enters by the ducts and is heated therein and in the chamber. During the heating process it is used for secondary combustion at the points where the furnace gases enter the chamber. In the cooling process the hot air enters the oven chamber at points in the upper part thereof and passes downwards over the charge, cooling as the charge cools. A. Cousen.

Manufacture of molybdenum compounds [abrasives]. Norske Molybdenprodukter A.-S. (G.P. 424,990, 15.5.21).—Molybdenum–sulphur compounds are heated with boron, titanium, or zirconium, or with mixtures of these, in such quantities that molybdenum boride, titanide, or zirconide is produced. Thallium, vanadium, or the metals of the iron and chromium groups, or closely allied metals, or compounds of these metals may also be added, and the hardness of the product can be increased by the addition of small quantities of carbon, silicon, nitrogen, or phosphorus, or compounds containing them. L. A. Coles.

Quartz working. E. R. Berry, Assr. to Gen. Electric Co. (U.S.P. 1,581,829, 20.4.26. Appl., 29.3.21).—See E.P. 191,463 ; B., 1923, 226 a.

Channel oven for ceramic and chemical purposes. H. T. Padelt (U.S.P. 1,583,739, 4.5.26. Appl., 27.6.25).—See E.P. 245,005 ; B., 1926, 275.

Method of treating ceramic mixtures. H. Spurrier (E.P. 242,916, 1.7.25).—See U.S.P. 1,559,652 ; B., 1926, 55.

Annealing glassware. R. L. Frink (E.P. 250,634, 13.12.24).

Burning ceramic wares. E. C. R. Marks. From A. C. Spark Plug Co. (E.P. 250,732, 16.3.25).

IX.—BUILDING MATERIALS.

Preparation of industrial sands. Weigel.—See I.

Patents.

Impregnation of wood. R. Ditmar (Austr. P. 100,729, 27.11.23).—Wood, particularly that for use in the manufacture of violins, organs, and pianos, is improved by steeping in rubber latex mixed with 0·1—10% of glycerin. L. A. Coles.

Device for burning cement, magnesite, lime, etc. H. Stehmann (U.S.P. 1,581,522, 20.4.26. Appl., 30.8.21).—See E.P. 188,424 ; B., 1923, 82 a.

Cement-roasting apparatus. I. E. Lanhoffer (U.S.P. 1,582,831, 27.4.26. Appl., 24.5.22).—See E.P. 195,585 ; B., 1923, 834 a.

Process of making vesicular products [building materials]. C. W. Boynton, Assr. to Government of the United States and to the People of the United States (U.S.P. 1,583,521, 4.5.26. Appl., 20.12.18).—See E.P. 153,030 ; B., 1920, 821 a.

Mixing composition for use with wood aggregates and structures. J. R. Garrow,

Assr. to NOVOCRETE CO. OF AMERICA, INC. (U.S.P. 1,583,713, 4.5.26. Appl., 25.9.23). See E.P. 220,677 ; B., 1925, 12.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Combustibility of coke and direct reduction in the blast furnace. W. W. HOLLINGS (Iron and Steel Inst., June, 1926. Advance proof. 9 pp.).—In discussing reduction in the blast furnace from the results of other workers, the author states that so far as the iron itself is concerned, Gruner's ideal—that the whole of the reduction should take place indirectly by means of carbon monoxide—should be fulfilled in practice. Direct reduction must involve a greater demand for heat from the furnace and so should be avoided. The only lines on which it would seem possible to make any considerable saving in the heat demands of a modern blast furnace would appear to be by a considerable enrichment of the blast with oxygen, whereby the rapidity of cooling of the ascending gases would be increased and the volume of gas per unit of carbon burnt reduced ; or by the substitution of pulverised quicklime blown in at the tuyères for limestone added at the top. This latter would save carbon which reacts with liberated carbon dioxide, and would conserve the heat required for this reaction and also the heat necessary for the dissociation of the limestone.

C. A. KING.

Chemical equilibrium of manganese, carbon, and phosphorus in the basic open-hearth process. C. H. HERTY, JUN. (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy. 26 pp.). Experimental data have been obtained from laboratory furnaces of $\frac{1}{2}$ lb. capacity and from 100-ton stationary open-hearth furnaces, the three fundamental principles taken into consideration in the work being the law of mass action, the van't Hoff isochore, and the distribution law of Nernst. Except when the slag contains more than 5% P_2O_5, the amount of residual manganese is controlled by the total manganese charged, the amount of this element volatilised, the volume, basicity, and iron oxide content of the slag, and the temperature. High-manganese charges increase the fluidity of the slag and the speed of working and increase the ratio of manganese sulphide to ferrous sulphide in the metal, resulting in a more complete elimination of ferrous sulphide by the final manganese addition. The solubility of ferrous oxide increases 1·25 times as fast as the solubility of manganese oxide (MnO) for a given rise of temperature. The quantity of iron oxide in the metal is not controlled by the residual manganese. Temperature and the concentration of iron oxide in the slag govern the carbon content of the metal and at 1482—1538° the equilibrium constant for the reaction $C+FeO=CO+Fe$ in the metal phase is 0·36. At the same temperature the solubility of carbon monoxide in pure iron is 0·018% by weight. The amount of phosphorus remaining in the metal depends on the total phosphorus in the charge, the temperature, and the volume, basicity, and iron oxide content of the slag. Equations are given showing the relationship between the variables controlling the phosphorus and those controlling the manganese.

M. COOK.

Catalytic action of iron ores in decomposing carbon monoxide. P. FARUP (Tidskr. Kem. Bergv., 1926, 6, 1—6, 15—17 ; Chem. Zentr., 1926, I., 2530—2531).—The catalytic action of various iron ores in powdered form, in lumps, and in the form of briquettes on the decomposition of carbon monoxide at 420° according to the equation $2CO \rightleftharpoons CO_2+C$ has been investigated. The results obtained were in all cases extremely erratic ; sometimes no action appeared to take place whilst at other times the reaction was strongly catalysed with the same sample of ore. In general briquetting appeared to accelerate the decomposition of carbon monoxide. The presence of much silica in the ore lowered considerably its catalytic action and addition of calcium carbonate or sodium chloride decreased the action to a slight extent, owing to the glazing of the surface.

A. R. POWELL.

Prevention of embrittlement in malleable cast-iron. L. H. MARSHALL (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy. 8 pp.).—Embrittlement of malleable cast-iron observed after galvanising is not due either to the pickling process or to the etching action of the molten zinc, as malleable iron itself heated to 460° and chilled becomes brittle. Higher impact values are obtained if malleable iron is quenched from 600—750°, and such heat-treated metal may be galvanised in the ordinary manner without becoming brittle. A further advantage of such heat-treatment is that malleable iron may be subjected to low temperatures (0°) without any lessening of shock resistance.

C. A. KING.

Report on the heterogeneity of steel ingots. (Iron and Steel Inst., June, 1926. Advance proof. 113 pp.).—A large number of ingots ranging from 15 cwt. to 172 tons in weight and, in general, chill cast, were examined along a section taken longitudinally through the central axis. Transverse sections were also made in certain cases. Photographs of the sections, after etching with various reagents, and sulphur prints were taken. Analyses of definitely located samples and scleroscope hardness determinations over the sections were carried out. The results so obtained indicate that sulphur, phosphorus, and carbon exhibit segregation, whilst silicon and manganese, which diffuse more readily in solid solution, do not show this phenomenon. In general there exists in an ingot an outer layer of ill-defined crystalline structure, due to the initial rapid cooling of the steel in contact with the mould. From this layer, a layer of columnar crystals grows inwards and upwards, the thickness of which is dependent on several factors. Proceeding towards the centre of the ingot, there is a region richer in segregates, which may occur in spheroids or thin strings. This zone of segregation is annular, its

inner boundary having the form of a truncated cone or pyramid, depending on the shape of the mould. Extending inwards from this zone, there is a central region, in the lower half of which the steel has a lower sulphur, phosphorus, and carbon content than in any other part of the ingot, while the composition of the upper portion approaches the average value for the steel; at the extreme top there is a region of maximum segregation. The axial portion of the ingot is always rather unsound.

L. M. Clark.

Distribution of silicates in steel ingots. J. H. S. Dickenson (Iron and Steel Inst., June, 1926. Advance proof, 20 pp.).—Slaggy matter in the form of small silicate particles occurs most abundantly in the central lower part of steel ingots, in which locality carbon, sulphur, and phosphorus are reduced to a minimum by segregation. This has been found to be the case in small as well as large ingots of carbon, nickel, and nickel–chromium steels both top-cast and bottom-cast. The slaggy matter, obtained as residues from complete solution of normal samples, scraped from external slag streaks, and from pipe cavities is of similar composition, containing large amounts of silica and manganese oxide together with varying amounts of ferrous oxide and alumina. In large ingots the silicate content of the first frozen skin at the bottom end is low and similar to that of the outer skin at the upper end, but it rises upwards and inwards to a maximum at or near the central axis within the lower part of the central pyramid of free crystals. The silicate globules from the region of high silicate content are larger than those from other portions of the ingot. It is suggested as a possible explanation of the high silicate region that the purer crystallites, of which there is a continuous downward movement to the narrowing and rising solid floor, intercept and carry down the coalesced particles of slaggy matter.

M. Cook.

Hardness of carbon steels at high temperatures. I. G. Slater and T. H. Turner (Iron and Steel Inst., June, 1926. Advance proof. 9 pp.).—The impact hardness of a series of carbon steels ranging from 0·02% to 1·105% C was determined at 50° intervals from 650° to 1200°. Little hardness was lost up to 650°, but between 650° and 900° all the steels softened considerably, the degree of softening increasing with the carbon content. Decrease in hardness was relatively less rapid above 900° and the hardness values of all the steels became more nearly equal. For riveting purposes the higher the carbon content up to the eutectoid composition, the more energy is required to shape the rivet, and there is a distinct advantage to be gained in performing rapid work on the rivet at the highest working temperature before the steel cools.

C. A. King.

Ratio of tensile strength of steel to the Brinell hardness number. R. H. Greaves and J. A. Jones (Iron and Steel Inst., June, 1926. Advance proof. 18 pp.).—The results of a large number of laboratory tests, under definite conditions, on several kinds of steels have been studied by statistical methods and the results of other investigators on the tensile strength–Brinell hardness number ratio have been examined. This ratio is dependent on the hardness of the material and on its yield ratio and to some extent on the composition and heat-treatment. It decreases with increase of yield ratio for a given class of steel and with increase of hardness up to 375 to 450 Brinell. The ratios recommended are 0·21, 0·215, and 0·22 respectively for heat-treated alloy steels with a Brinell hardness of 250—400, heat-treated carbon steels and alloy steels with a hardness below 250, and medium carbon steels as rolled, normalised, or annealed. The value 0·23 is suitable for mild steels as rolled, normalised, or annealed. None of these ratios is applicable to austenitic or severely cold-drawn steels. Owing to the want of uniformity in a piece of steel and consequent variations in the Brinell figures the probable error of the ratio for a given series of steels is rarely less than ±0·003.

M. Cook.

Ghost lines and the banded structure of rolled and forged mild steels. J. H. Whiteley (Iron and Steel Inst., June, 1926. Advance proof. 6 pp.).—Only when the variation in phosphorus content between two adjacent areas exceeds 0·07% can it cause removal of carbon, between the Ar3 and Ar1 points, from the richer area. In most cases it is still doubtful whether this heterogeneity is the cause of ghosts, since other factors may influence the movement of carbon to an extent which outweighs the influence of phosphorus. Banded structures have been observed in steels containing phosphorus as low as 0·004%, and it is shown that in certain cases carbon may move from one region to another higher in phosphorus. Ziegler's theory that non-metallic inclusions act as nuclei for the crystallisation of ferrite and so produce a ghost formation does not adequately account for the facts which are given.

M. Cook.

Effects of arsenic on steel. A. E. Cameron and G. B. Waterhouse (Iron and Steel Inst., June, 1926. Advance proof. 20 pp.).—Steels containing carbon about 0·5% and having similar compositions with regard to sulphur, phosphorus, manganese, and silicon, with arsenic contents varying from 0·20 to 0·46% in the molten state, exhibit segregation of arsenic on solidification, since the arsenic content of the billets obtained by cropping off the ends of the ingots falls in each case to approximately 0·2%. It is concluded that an equilibrium exists between the arsenic and carbon, which tends to limit the arsenic content to 0·2% in 0·5% carbon steels. Indications of red-shortness are obtained during the forging of these steels, and this is ascribed to the presence of arsenic, since no red-shortness is apparent in a control sample of steel free from arsenic. The position of the transformation points of the 0·5% carbon steel is not materially modified by arsenic (0·22%), while a slight increase in tensile strength and a small but rather greater decrease in ductility result. There is a considerable reduction

in the shock-resisting capacity of these steels as measured by the Charpy impact test, and this effect is most noticeable when the metal is in a hardened condition. No effect on the corrosibility or on the case-hardening properties is caused by the presence of arsenic up to 0·22% and the steels can be welded if a suitable welding compound be used. Microscopical examination shows that arsenic segregates, forming a banded structure which persists throughout all the heat treatments applied, and it is concluded that arsenic does not diffuse in γ-solid solution. No evidence was obtained for the existence of a brittle eutectic of iron–iron arsenide. Arsenic occurs in steel only as a solid solution of arsenic in iron. L. M. Clark.

Effect of mass in the heat treatment of nickel steel. W. Rosenhain, R. G. Batson, and N. P. Tucker (Iron and Steel Inst., June, 1926. Advance proof. 33 pp.).—Results of tests in which round bars of nickel steel (about 3·5% Ni) of 2, 4, 7, and 10 in. diam. were normalised at 850°, oil-hardened at 850°, and oil-hardened and quenched at temperatures between 300° and 700°, showed that the quenching effect was insufficient to effect complete hardening of the steel even in pieces 2 in. in diameter. In this size the strength after quenching was only about 60 tons per sq. in., rising to 80 tons on tempering at 300°, and then falling almost to the strength of normalised material when tempered at 650°. A bar of 4 in. diam. behaved somewhat similarly and the strength of the 7 in. and 10 in. diam. bars was not increased by tempering the hardened pieces unless the temperature exceeded 500°. The largest size was little affected at the centre by quenching. Quenched nickel steel possessed a low notched-bar figure compared with normalised steel, but tempering at high temperatures, *e.g.*, 600° and 650°, caused a considerable improvement even in the 7 in. and 10 in. pieces. The effect of heat treatment on the structure of the steel was marked in so far as the outer portions of the material were concerned but the centre of a 10 in. bar was little different in microstructure or mechanical properties from the normalised steel. C. A. King.

Hardening and tempering of high-speed steel. A. R. Page (Iron and Steel Inst., June, 1926. Advance proof. 24 pp.).—The conditions of hardening and tempering two high-speed steels, J1 and J2, of the composition C 0·55—0·65% (J2, 0·7—0·8%) Si 0·2%, W 13—14%, Cr 3—4%, Mn 0·4—0·5%, V 0·25—0·5%, S 0·05%, P 0·06% were investigated. J1 was hardened completely by soaking for 10 min. at 1250°, or at 1300° for 2 min., though at the latter temperature there was enormous crystal growth if the time of soaking was extended. The higher carbon content of J2 reduced the temperature required for correct hardening to 1150°, but the range of temperature and time for hardening were greater than for J1. Excessive brittleness in the hardened material due to the production of an intercrystalline eutectic constituent, evidently caused by incipient fusion at the crystal boundaries, was more marked in the higher carbon steel. The hardened steels after tempering at 600° for 2 min. appeared perfectly stable as regards hardness at any temperature up to 600°, but the hardness and appearance of the fracture in the hardened state were no criteria of the production of the red-hardness property. C. A. King.

Advances in high-speed alloys. E. H. Schulz, W. Jenge, and F. Bauerfeld (Z. Metallk., 1926, 18, 155—158).—The effect of heat treatment on the structure and magnetic properties of a cobalt magnet-steel containing 16% Co, 9% Cr, 2·5% Mo, and 1% C has been investigated. This alloy in the soft annealed state has a sorbitic structure with large white inclusions of carbide ; it exhibits arrest points on heating at 600° and 820°, and on cooling at 775° and 590°, the higher points corresponding with the Ac1 and Ar1 points of steel respectively. The usual heat treatment for developing the magnetic properties comprises three steps : (i) heating for 5 min. at 1150—1200° followed by cooling in air, whereby a homogeneous austenitic structure is produced ; (ii) heating to 750° followed by slow cooling to 500° in the furnace and to the ordinary temperature in air, whereby the austenite is decomposed partially with the precipitation of finely-divided carbide ; (iii) heating to 1000° followed by air-cooling, whereby the carbide redissolves in the α-iron forming hardenite. The changes in hardness and magnetic properties undergone by the metal in this treatment are shown in the following table :—

	Brinell hardness.	Remanence.	Coercivity.	Electrical conductivity.
Annealed ..	341	12,600	35	2315
after (i) ..	218	1500	—	1482
„ (ii) ..	394	12,000	49	1919
„ (iii) ..	700	8000	190—205	1357

The structure of stellite consists of a groundmass of eutectic nature containing needles of carbide. The smaller the needles the greater is the cutting power of the alloy ; to obtain this structure the alloy should be cast in as thin plates as possible so that cooling is relatively rapid. In this form the alloy is suitable for making draw-rings for steel shells, having a life 250—1000 times as long as that of similar rings of cast iron, besides being non-rusting. Corrosion tests over 4 months showed that stellite alloys lose only 0·07% in weight in 20% nitric acid and 0·3% in 50% sulphuric acid and are not attacked by saturated solutions of sodium chloride or sulphide, 50% solutions of sodium hydroxide, or glacial acetic acid. A. R. Powell.

Determination of carbon in pig-iron, steel, and ferro-alloys. E. Schiffer (Stahl u. Eisen, 1926, **46**, 461—468 ; cf. van Royen, B., 1924, 425).—The mixture of carbon dioxide and oxygen obtained by burning ferrous materials in the minimum quantity of oxygen at 1100—1200° is collected. The volume of carbon dioxide absorbed by potassium hydroxide solution enables the carbon content of the material to be determined. The method is satisfactory for steels and pig-irons containing little sulphur and having a carbon content of not more than 1%,

especially when lead oxide is used as a flux. It is not satisfactory with ferrous alloys in general. The speed with which the determination can be made renders this method valuable for controlling smelting processes. Alternatively, the gas mixture may be passed through barium or sodium hydroxide solution. Titration of the excess of alkali gives the amount of carbon dioxide formed in the combustion.
L. M. CLARK.

Determination of phosphorus in steels containing tungsten. T. E. ROONEY and L. M. CLARK (Iron and Steel Inst., June, 1926. Advance proof. 6 pp.; cf. A., 1923, ii, 251).—Confirmation is given for the observation of previous investigators that, when tungsten steels are decomposed by treatment with acid, some phosphorus is contained in the iron solution whilst the remainder is retained in the insoluble tungsten residue. Previously described methods for the determination of phosphorus in such steels are unsatisfactory. A newly-devised method gives trustworthy results. The steel is digested with *aqua regia* until the iron is dissolved and the tungsten is converted into tungstic oxide. After evaporation, the solid residue is heated to drive off nitric acid and the ferric oxide is dissolved out with hydrochloric acid. The tungstic oxide containing some phosphorus is dissolved in ammonia and a little ferric chloride is added to the solution, which is then made slightly acid with hydrochloric acid. On precipitation of the iron from this solution with ammonia, the phosphorus is carried down with the ferric hydroxide. The precipitate is dissolved in hydrochloric acid, reprecipitated with ammonia, and redissolved in hydrochloric acid. This solution is added to the main bulk of ferric chloride solution from which hydrochloric acid is expelled by evaporation with nitric acid. Phosphorus is then determined by precipitation as phosphomolybdate and titration with alkali. The method is applicable to steels containing vanadium. L. M. CLARK.

H-ion concentration [of water] and corrosion. J. R. BAYLIS (Eng. and Cont., Water Works Issue, 1925, 64, 1279—1280).—A factor in corrosion is the effect of p_H on oxidation rates, soluble iron in water containing excess oxygen oxidising much more rapidly at p_H 9 than at p_H 6. A large number of experiments on the amount of iron which will remain in solution at different p_H values gave the following results: p_H 9·0, less than 0·1 p.p.m.; p_H 8·0, 0·1 p.p.m.; p_H 7·5, 1·0 p.p.m.; p_H 7·0, 4 p.p.m. Below p_H 7·0 the soluble iron content increased rapidly. There is a natural tendency for tubercles to form an impervious coating near the surface exposed to the water, where the soluble iron from the interior comes in contact with dissolved oxygen. If the water is slightly acid, say p_H 6·0, the rate of oxidation is low and there is a tendency for the soluble iron to diffuse into the water. If the water is alkaline, about p_H 8·5, nearly all the soluble iron will be precipitated at the surface of the tubercle and in a few months will form an impervious coating of a crystalline iron oxide. If alkalinity is due to calcium carbonate or bicarbonate, and p_H is adjusted to a point which favours precipitation of calcium carbonate, this will also aid greatly in preventing corrosion, in addition to the natural tendency of corroding iron to protect itself in alkaline water. Pits have been found to be concentration cells in which negative ions, *e.g.*, chloride and sulphate, are concentrated. The solution in the interior of an active pit has a p_H close to 6·0, regardless of the H-ion concentration of the surrounding solution. R. E. THOMPSON.

Factors other than dissolved oxygen influencing the corrosion of iron pipes. J. R. BAYLIS (Ind. Eng. Chem., 1926, 18, 370—380).—Dissolved oxygen is the governing factor in the corrosion of new iron water pipes by town water only until the pipe becomes coated with a film of rust. After this other factors chiefly connected with the composition of the water become of primary importance. Ferrous hydroxide is the first product of corrosion; its solubility in the water depends on the p_H, and with a p_H above 8 and in the absence of dissolved oxygen, it is almost insoluble in natural waters. Ferrous carbonate is only very slightly soluble in water saturated with calcium carbonate, but its solubility increases rapidly as the p_H falls below calcium carbonate equilibrium. In waters having a high p_H the final product of the reaction between iron and the dissolved carbonates is a very insoluble ferrous carbonate. No gaseous hydrogen is liberated from pure distilled water by iron even after several months but the presence of negative ions other than OH′ ions causes hydrogen to be evolved relatively rapidly. Thus, addition of calcium sulphate or sodium chloride to air-free distilled water in which are placed some iron filings results in hydrogen evolution after a few days and an increase in the p_H of the water to about 9; similar results are obtained on the addition of calcium hydrogen carbonate, and it therefore appears that this reaction is of fundamental importance in the corrosion of iron water pipes, for, unless it takes place, the surface of the metal becomes coated with a passive film of ferrous carbonate which is only extremely slowly oxidised by the dissolved oxygen. Some of the corrosion products occurring in water pipes are very magnetic and attach themselves to the surface of the metal, forming a very porous, fibrous aggregate. These deposits do not cause pitting, as the corrosion products readily diffuse from them. When, however, the rust coating becomes so thick that diffusion of soluble iron salts from inside the coating is prevented the water just outside the deposit still retains its content of dissolved oxygen and the corrosion products are precipitated inside the coating, thus soon rendering it impervious, and a pit is formed. This will continue to increase in size as long as the p_H of the water inside the membrane of rust is about 6, *i.e.*, as long as the water retains iron salts (sulphates or chlorides) in solution. When the p_H rises above 6 action ceases and the pit becomes dormant. In acid waters the covering membrane has a great tendency to break; when this occurs the whole process is repeated, so that after a time these

membranes become of considerable size. In alkaline waters breakage occurs only rarely and the pipes therefore have a longer life. A. R. POWELL.

[Silver–gold] parting plant at the U.S.S. Lead Refinery Inc. F. F. COLCORD (Trans. Amer. Electrochem. Soc., 1926, **49**, 229—238. Advance copy).—Doré containing 95—98% Ag and 1—5% Au, with small amounts of lead, copper, bismuth, selenium, and tellurium, is refined electrolytically in horizontal cells at the U.S.S. Lead Refinery. The electrolyte contains 50 g. of silver and 60 g. of copper (as nitrates) per litre; the cathode consists of a graphite plate lying on the bottom of a stoneware cell, 52×24×9 in., and the anodes of thin ingots of doré inside a wooden framework sliding on the top of the cell and covered with cotton duck outside and muslin inside. A current density of 40 amp. per sq. ft. of anode surface at 3 volts is used in the electrolysis and from time to time the silver crystals are scooped out from the bottom of the cell with a perforated aluminium shovel. The gold slime from the muslin linings is periodically collected, washed to remove soluble salts, and boiled with sulphuric acid in an "ironac" pan. The resulting gold sponge when melted into bars is 995 fine. The silver sulphate solution is treated with copper to precipitate the silver, which is melted in a Faber du Faur furnace. Photographs and plans of the plant showing working details are given. A. R. POWELL.

Conductivity of electrolytes used in the electrolytic separation of silver and gold. F. F. COLCORD, E. F. KERN, and J. J. MULLIGAN (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy. 9 pp.).—The conductivity of the electrolyte increases with the copper and silver content; copper is more effective than silver, one unit raising the conductivity to the same extent as 1½ units of silver in electrolytes containing 30—60 g. per litre each of silver and copper. Ammonium nitrate increases the conductivity, the maximum effect being shown when it is present to the extent of 165 g. (=35 g. of ammonia) per litre. The deposited silver is of a fine crystalline nature when the silver and copper content of the electrolyte is low (40 g. per litre) and with very low silver content (35 g. per litre), the crystals are either finer or they assume a needle-like shape. In the presence of copper the crystals become coarser as the silver increases up to 60 g. per litre, and increasing copper to 80 g. per litre results in a coarse and more compact deposit. Ammonium nitrate causes the formation of a more compact deposit from electrolytes containing between 40 and 60 g. per litre each of silver and copper. M. COOK.

Electrodeposition of zinc from electrolytes containing gelatin and aluminium sulphate. P. K. FRÖLICH (Trans. Amer. Electrochem. Soc., 1926, **49**, 285—302. Advance copy).—The electrodeposition of zinc from chloride and sulphate solutions of varying degrees of acidity with and without the addition of gelatin and aluminium sulphate has been investigated. Gelatin invariably causes a dark, rough and nodular deposit to be obtained, whilst the cathode polarisation and to a smaller extent the anode polarisation increase with increasing acidity. From this and other data it appears that there is a thin film of electrolyte on the surface of the cathode having a p_H above 5, while the p_H of the main solution is less than 5; hence the gelatin is positively charged and basic in character in the body of the electrolyte and negatively charged and acidic in character on the inner surface of the film owing to its inversion in passing through the isoelectric point, p_H 4·7. The action of aluminium sulphate is similar to that of gelatin in causing poor deposits of zinc, probably owing to precipitation of colloidal aluminium hydroxide in the cathode film. This would explain the difference in the action of gelatin and aluminium sulphate in the electrodeposition of copper and of zinc. A. R. POWELL.

Properties of sand-cast alloys of aluminium containing silicon and magnesium. S. DANIELS (Ind. Eng. Chem., 1926, **18**, 393—398; cf. B., 1925, 73, 506).—Tensile, hardness, and density determinations were carried out on aluminium alloys containing 0·5—0·6% Mg, and silicon in amounts increasing from 0·27 to 4·67%. For sand-cast specimens containing up to 1% Si, the ultimate strength is about 16,000 lb./sq. in., and rises to 20,000 at 3—4·7% Si. The Brinell hardness increases similarly, but the elongation and ductility are markedly diminished. The properties are greatly improved by heat treatment for 96 hrs. at 550°, quenching from this temperature, followed by ageing for 8 hrs. at 150°, after which alloys with more than 0·3% Si showed a uniform ultimate strength of about 30,000 lb./sq. in., whilst the ductility was increased compared with the sand-cast state. The Brinell hardness also increased, but the density was not affected by heat treatment. Alloys containing 0·27% Si were little affected by heat treatment because the silicon content is less than that required to convert all the magnesium into the compound Mg_2Si; the quantity required for this purpose is not that given by the formula but is modified by the presence of iron or other impurities. The microstructure of the alloys is described. W. HUME-ROTHERY.

Electrochemical and *X*-ray studies of lead deposits. P. K. FRÖLICH, G. L. CLARK, and R. H. ABORN (Trans. Amer. Electrochem. Soc. 1926, **49**, 239—260. Advance copy).—Lead electro-deposits from acetate, nitrate, perchlorate, fluoborate, and fluosilicate electrolytes, in the presence and absence of free acid and of gelatin, have been investigated by *X*-ray analysis and potential measurements. The best deposits are given from solutions of perchlorate with excess of both acid and gelatin, when maximum cathodic polarisation, minimum grain size, absence of dendrites, but evidence of preferred orientation were observed. Small grain size is favoured by high acidity and current density and by the presence of gelatin. The effect of the base metal of an iron cathode is small because no penetration takes place, but distinct differences were observed between waxed and unwaxed platinum cathodes where the lead

penetrates the platinum surface. In contrast to other metals, increased current density favours preferred orientation of the crystal grains, and the data indicate that the (211) planes are parallel to the electrode surface. Preferred orientation is favoured by small grain size and hence by the addition of gelatin. Grain size increases with the thickness of the deposit, and hence the outer layers show less preferred orientation than the innermost. The different electrolytes in the presence of free acid but with no gelatin give increasing grain size in the order, fluosilicate, perchlorate, and fluoborate. The polarisation curves indicate that the resistance met with in the cathodic deposition of lead crystals is small. The equilibrium potential of lead in *N*-nitrate solution was determined as −0·158 volt at 24—25°. A Laue diagram of a lead tree or dendrite shows that it is not one single crystal, but is built up in orderly fashion, with a certain amount of internal strain.
W. HUME-ROTHERY.

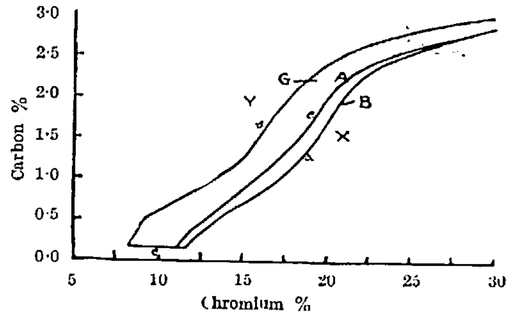
Reaction $R_mS_n + 2nSO_2 = R_m(SO_4)_n + nS_2$. Chemistry of the roasting of some ores. J. MILBAUER and J. TUCEK (Chem.-Ztg., 1926, 50, 323—325).—With the exception of mercuric sulphide which volatilises unchanged, the natural and artificial sulphides of the metals react at high temperatures with sulphur dioxide according to the equation $R_mS_n+2nSO_2=R_m(SO_4)_n+nS_2$. With sulphides of the alkalis and alkaline earths sulphates are the sole products but with other metal sulphides various side-reactions take place; with the sulphides of copper, lead, bismuth, and antimony large amounts of metal are formed by the interaction of unchanged sulphide with the sulphate first formed. The reaction in the case of the sulphides of aluminium, chromium, zinc, manganese, iron, cobalt, nickel, tin, and cadmium begins only at temperatures at which the sulphate is unstable, so that the final product is the oxide.
A. R. POWELL.

See also A., May, 459, **Determination of orientation of crystallites in metallic conglomerates** (TAMMANN and MÜLLER); ***X*-Ray investigations of structure of some alloys** (HOLGERSSON). 460, **Crystal structure of Heusler alloys** (HARANG). 461, **Temperature coefficient of magnetic permeability of sheet steel** (SPOONER); **Sublimation and crystallisation of metals** (PECZALSKI). 462, **Polymorphism of iron** (BREDEMEIER). 463, **Free energy and heat of formation of iron carbide between 650° and 700°** (MAXWELL and HAYES). 466, **Relation of ferrous metals** (FLETCHER). 474, **Amalgams of gold** (BRITTON and MCBAIN). 475, **System iron–tin** (WEVER and REINECKEN). 482, **Speed of dissolution of copper in aqueous ferric chloride** (BEKIER and TRZECIAK); **Metals and nitric acid** (KLEMENC).

Influence of segregation on corrosion of boiler tubes and superheaters. WOODVINE and ROBERTS.—See I.

PATENTS.

Article [chromium steel] resistant to food acids and process of making it. P. A. E. ARMSTRONG, Assr. to LUDLUM STEEL CO. (U.S.P. 1,554,615, 22.9.25. Appl., 1.12.22).—Chromium steels containing chromium and carbon corresponding



to the co-ordinates of any point within an area A bounded by lines, *a*, *b*, *c*, *d* on the graph are hardened and rendered immune to food acids by heating them to 24—300° above their upper transformation point and cooling rapidly.
C. A. KING.

Protection of metals [iron] against corrosion. I. T. BENNETT, Assr. to T. E. MURRAY (U.S.P. 1,578,254, 30.3.26. Appl., 26.6.24).—The surface of iron or steel is coated first with chromium and then with another protective coating, *e.g.*, nickel.
C. A. KING.

Treatment of ores or other materials with liquids. A. RAMÉN (E.P. 237,571, 18.6.25. Conv., 23.7.24).—For the moistening of roasted ore previous to extraction, the material is introduced in a thin layer on to the bottom of a closed container from a feeding hopper, the outlet of which extends close to the bottom of the container. The mouthpiece of the hopper is capable of rotation so as to distribute the material in a circle on the bottom of the container, whence, after wetting, it is discharged by a scraper through an outlet. The liquor pipe may be fitted inside the feeding hopper and extends nearly to the bottom of the container. In another form the container is provided with a raised conical middle part which is surrounded by a circular gutter. Ore fed in spreads into the gutter in which it is wetted and then is removed by a scraper.
C. A. KING.

Electrolytic production of zinc from ores. S. FIELD, F. PETERSSON, W. E. HARRIS, and METALS EXTRACTION CORP., LTD. (E.P. [A] 249,764 and [B] 249,609, 29.12.24).—(A) Zinc sulphide ores containing iron and manganese are roasted at 650° until the sulphur content is reduced to a point at which steam does not interfere with the oxidation of the sulphur (about 9% S) and the roasting is continued in an atmosphere of steam and air to prevent the formation of ferrites and manganites and to convert the maximum amount of iron into the magnetic oxide. (B) The roasted ore is crushed and introduced, a little at a time, into dilute sulphuric acid at 85—90° until the acidity, after stirring, is reduced to 0·2%. The hot solution is then completely neutralised with a small amount of high-grade zinc residues and agitated with a small amount of calcium carbonate which flocculates the colloidal silica. To oxidise any

small amount of ferrous sulphate present a quantity of anode mud (chiefly lead dioxide) is then added and agitation continued until all the iron is precipitated. The solution is filtered hot and the filtrate passed to a second vessel in which it is further purified according to E.P. 162,030 (B., 1921, 436 A). A. R. POWELL.

Metal coatings on aluminium and aluminium alloys.—B. JIROTKA and F. CONSTEN (E.P. 249,971, 5.2.25).—A metal coating is obtained on aluminium without preliminary freeing from grease and the use of cyanide compounds by immersion in a bath of hot alkaline solution containing salts, other than cyanides or fluorides, of copper, nickel, chromium, or other heavy metal, and potassium carbonate and bicarbonate alone or mixed with the corresponding sodium salts. Compounds which liberate oxygen, *e.g.*, soluble chromates, may be added to the bath. The addition of 10—20% of glycerin to the bath produces brighter coatings. A suitable bath contains 25 g. of crystalline copper sulphate, 25 g. of potassium carbonate, 25 g. of sodium bicarbonate, and 10 g. of potassium dichromate in 2·5 litres of water. M. COOK.

Light aluminium alloy.—V. E. HYBINETTE (U.S.P. 1,579,481, 6.4.26. Appl., 22.1.25).—An alloy containing more than 90% Al, 0·50 to 3·0% Ni, and from 0·25 to 1·50% of metals of the chromium group is claimed. M. COOK.

Bearing material. H. M. WILLIAMS, Assr. to GEN. MOTORS RESEARCH CORP. (U.S.P. 1,556,658, 13.10.25. Appl., 24.3.22).—A mixture of finely-divided copper 90 pts. and tin 20 pts., together with 6% of graphite, is compressed under a pressure of 40,000—100,000 lb. per sq. in., then packed in lampblack and heated in a sealed container for 5—7 hrs. at 590—760°. The finished bearing metal will absorb up to 8% of oil.

Metallic composition [lead amalgam]. A. MILLER (U.S.P. 1,570,763, 26.1.26. Appl., 19.7.24).—A metallic composition containing 90% Pb and 10% Hg is claimed. It is specially suitable for storage battery plates. It has a greater porosity than lead, is rigid, practically non-corrosive, and has a high electrical conductivity. M. COOK.

Melting metals. Y. A. DYER (U.S.P. 1,578,648, 30.3.26. Appl., 5.5.25).—Carbonaceous material is burned in a combustion chamber in the presence of air which is preheated to 315° but not exceeding 1040° by passing it through pipes which form the dome of the chamber. The resulting hot gases, after mixing with additional air, are introduced at a pressure of ¼ to 1½ lb. per sq. in. into a second chamber and through the charge which consists of the metal to be melted and carbonaceous matter in alternate layers. M. COOK.

Metallurgical process [for recovering copper from ores]. W. E. GREENAWALT (U.S.P. 1,579,356, 6.4.26. Appl., 27.1.25).—Roasted copper ore is agitated with an acid solution to extract a portion of the copper, and the rich liquor is separated from the insoluble residue, which is then treated to separate the slimes from the sand. The copper solution is reduced with sulphur dioxide, treated with copper sulphide precipitated from weak solutions, and finally electrolysed for the deposition of copper and regeneration of acid. This acid liquor is then used for extracting the remainder of the copper from the sands by percolation. A. R. POWELL.

Separation of copper and bismuth from molybdenite. E. POKORNY (G.P. 425,364, 16.6.23).—Molybdenite is treated with chlorine in the presence of water, preferably at a raised temperature, until the copper and bismuth are completely dissolved, or, alternatively, the ore is treated with chlorine until the copper and bismuth particles have been attacked superficially, and is then subjected to a flotation process. L. A. COLES.

Protecting metallic articles against oxidation. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of G. H. HOWE (E.P. 224,534, 5.11.24. Conv., 6.11.23).—See U.S.P. 1,555,578; B., 1925, 996.

Producing an adherent coating of an oxygenous magnesium compound [on magnesium or its alloys]. C. B. BACKER (Reissue 16,340, 4.5.26, of U.S.P. 1,451,755, 17.4.23. Appl., 29.1.25).—See B., 1923, 507 A.

Treating metals to inhibit excessive grain growth. G. L. KELLEY, Assr. to E. G. BUDD MANUF. CO. (U.S.P. 1,581,269, 20.4.26. Appl., 23.4.24).—See E.P. 248,801; B., 1926, 411.

Treating impure molten metals. H. HARRIS (U.S.P. 1,582,037—8, 27.4.26. Appl., 26.4.24).—See E.P. 217,391; B., 1924, 679.

Sulphating process for ores and concentrates. J. B. READ and M. F. COOLBAUGH, Assrs. to COMPLEX ORES RECOVERIES CO. (U.S.P. 1,582,347, 27.4.26. Appl., 26.11.19).—See E.P. 200,852; B., 1923, 1077 A.

Magnetic alloy. W. S. SMITH, N. POPPLEFORD, and H. J. GARNETT (U.S.P. 1,582,353, 27.4.26. Appl., 2.5.25).—See E.P. 224,972; B., 1925, 76.

Method of coating vessels [with an alloy]. M. DREIFUSS (U.S.P. 1,582,668, 27.4.26. Appl., 14.11.23).—See E.P. 220,791; B., 1924, 875.

Method of treating aluminium-silicon alloys. A. G. C. GWYER and H. W. L. PHILLIPS, Assrs. to BRITISH ALUMINIUM CO., LTD. (U.S.P. 1,583,549, 4.5.26. Appl., 9.6.25).—See E.P. 219,346; B., 1924, 793.

Modifying ignition temperature of carbonaceous materials (U.S.P. 1,576,179).—See II.

High-frequency electric furnaces (E.P. 245,414).—See XI.

Arc welding electrodes (U.S.P. 246,140).—See XI.

XI.—ELECTROTECHNICS.

Insoluble anodes for the electrolyses of brine. C. G. Fink and L. C. Pan (Trans. Amer. Electrochem. Soc., 1926, 49, 183—228. Advance copy).—Graphite, platinum, magnetite, carborundum, ferrosilicon, lead peroxide, manganese dioxide, and natural anthracite were found to be not entirely satisfactory in use. Although the rate of anodic corrosion of pure silver, lead, or mercury is very high, the rate and with it the cell voltage drop instantly upon the addition of a small percentage of silver to lead or *vice versa*. A silver-lead anode containing 61% Ag best fulfils the requirements of an anode material for the electrolysis of brine. The sharp reduction in anodic corrosion is due to the formation of a protective film over the surface of the anode. With a properly formed film, the anode does not appreciably corrode or lose weight in the anolyte, nor is it attacked by chlorine providing the cell is not on open circuit. The silver-lead alloy has a low electrode potential and low chlorine overvoltage, is easily cast and machined, is mechanically strong, ductile, malleable, and has a low melting point. The anodes even when dissolved, do not contaminate the products of the brine cell. They have a high specific electrical conductivity compared with graphite and can be "burned" or welded so as to eliminate a contact resistance entirely. Their initial cost is higher than that of graphite anodes but their maintenance cost is lower than for either the graphite or platinum anodes used in some types of cells. A small amount of mercury or manganese in silver-lead anodes increases the corrodibility of the anode in brine solutions. An alkali-chlorine cell using a silver-lead anode is described. J. S. G. Thomas.

Simple method of measuring polarisation and resistance [of electrolytes]. H. E. Haring (Trans. Amer. Electrochem. Soc., 1926, 49, 31—46. Advance copy).—The polarisation at an electrode during electrodeposition may be measured by determining the potential difference between the electrode and an auxiliary electrode of the same metal in the same electrolyte. The auxiliary-electrode vessel consists of a glass tube with a capillary extension which is bent at right angles near one end. The capillary tip is either kept in contact with the main electrode or else kept at a constant distance; in each case a correction for the potential drop is necessary, though in the former arrangement its magnitude is uncertain. A rod of the metal dips into the tube; replacement of the rod by a gauze spiral or the introduction of a high resistance in the auxiliary circuit eliminates any small polarisation which may be produced at the auxiliary electrode. A permanent cell for polarisation and resistance measurements is described. The cathode and anode are placed at the ends of a rectangular vessel which is divided into three equal compartments by means of rectangular partitions of coarse gauze. The latter are shown to act as auxiliary unpolarised electrodes and the above considerations apply. A measurement of the potential drop between the partitions (with a potentiometer or a high-resistance voltmeter, the maximum reading always being taken) enables the resistance of the electrolyte to be calculated, the current being known. The method was tested with three typical plating solutions; the accuracy approached that of the usual methods. The results of the tests are represented graphically. A device by means of which a calomel electrode can be used sa the auxiliary electrode is described. S. K. Tweedy.

Secondary reaction in the lead accumulator. C. Féry with C. Chéneveau (Bull. Soc. chim., 1926, [iv], 39, 603—612).—See B., 1925, 997; 1926, 412.

Influence of alternating currents on electrolytic corrosion of iron. A. J. Allmand and R. H. D. Barklie (Trans. Faraday Soc., 1926, **22**, 34—45).—See B., 1926, 277.

See also A., May, 479, **Theory of overvoltage** (Knobel); **Electrolysis of acid solutions of copper sulphate** (Burt-Gerrans), 483, **Electrolytic separation of metals** (Brown); **Electrolysis of electrolytes containing cuprous chloride** (Hänsel). 509, **Electrochemical oxidation of aromatic hydrocarbons containing nuclear chlorine** (Fichter and Adler).

See also pages 475, **Oils for transformers etc.** (Baum). 485, **Nitrogen fixation** (Tartar and Perkins). 494, **Conductivity of electrolytes** (Colcord and others); **Silver-gold parting** (Colcord); **Electrodeposition of zinc** (Frölich). 503, **Dielectric constant etc. of rubber and guttapercha** (Curtis and McPherson).

Patents.

High-frequency electric furnaces. Westinghouse Lamp Co., Assees. of H. C. Rentschler and J. W. Marden (E.P. 245,414, 3.1.25. Conv., 5.1.25).—In a high-frequency electric induction furnace for heating rare metals, a support consisting of a crucible or disc for the material to be heated is located within an induction coil, the support being composed of a rare metal compound, *e.g.* thoria, which does not dissociate at high temperatures and which will not contaminate the metal being heated. J. S. G. Thomas.

High-frequency dielectric and magnetic furnace. C. T. Allcutt, Assr. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,572,873, 16.2.26. Appl., 29.12.23).—The furnace consists of a crucible, surrounded by a coil connected to a source of high-frequency alternating current, and placed between two electrodes also connected to a source of high-frequency alternating current. It is used to heat substances, such as metal oxides, which have a relatively high negative temperature coefficient of resistivity. The substance is first heated by the action of the alternating electric field set up by current applied to the electrodes, and when sufficiently hot to have its initial resistivity greatly reduced it is rapidly heated by the alternating electromagnetic field generated by passing an alternating current through the coil. If the substance to be heated is

a conductor even when cold, the crucible is made of an imperfect dielectric, such as glass or quartz, and is initially heated by the electric field. The apparatus is also of value in heating articles such as vacuum bulbs which comprise a metallic conductor and an imperfect dielectric such as glass. The former is heated by the electromagnetic field and the latter by the electric field. T. S. WHEELER.

Electric rotating resistance furnace. F. ANDERSEN, Assr. to TROLLHÄTTANS ELEKTROTHERMISKA AKTIEBOLAG (U.S.P. 1,576,621, 16.3.26. Appl., 22.3.23).—In a resistor for electric furnaces, the specific electrical resistance of the material constituting the current-supplying parts is lower than that of the material constituting the intermediate part of the resistor, in the proportion of at least 1 : 2. M. E. NOTTAGE.

Arc-welding electrodes. BRITISH THOMSON-HOUSTON CO., Assees. of J. M. WEED (E.P. 246,140, 12.1.26. Conv., 13.1.25).—For use with an iron electrode for arc welding, a flux containing sodium titanate and finely-divided metallic zinc is claimed. The method of compounding the electrode may vary, *e.g.*, the iron core may be galvanised and then coated with a flux containing sodium titanate, or an iron core may be inserted in a galvanised iron sheath of higher m.p. than the core, the intervening space being occupied by the flux. C. A. KING.

Separating light materials from gases. H. A. WINTERMUTE, Assr. to RESEARCH CORP. (U.S.P. 1,579,462, 6.4.26. Appl., 11.2.25).—A gas stream is subjected to the action of an electric field, the velocity of the stream being such that masses of material deposited from the gas by the field are detached from the walls of the conduit in agglomerated form and carried forward by the stream. J. S. G. THOMAS.

Electrical purification of gases by means of alternating current. H. ROHMANN, and ELEKTRISCHE GASREINIGUNGS-GES.M.B.H. (G.P. 425,026, 30.10.21).—In plant for the electrical purification of gases by means of alternating current, in which dust particles carried by the gas stream are first electrically charged by passage between high-tension discharge electrodes and subsequently precipitated on electrodes at lower potential, a phase displacement of such magnitude as to produce the maximum efficiency of precipitation is introduced between the respective potentials employed. J. S. G. THOMAS.

Electrical purification of gases. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 425,039, 15.10.22).—Gas is caused to flow helically and turbulently through a tube wherein it is subjected to the high-tension discharge from an upright conductor arranged along the axis of the tube. The direction of rotation of the gas in the tube is chosen to correspond with the direction of electric lines of force surrounding the conductor. Each period of gas purification is followed by a period during which dust particles clinging to the sides of the tube are removed by means of a sand blast blown into the tube so as to circulate in the same direction as the gas stream. If desired, dried sand or similar material may be added to the gas during the process. J. S. G. THOMAS.

Discharge electrode for electrical gas purification. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 425,273, 29.8.23).—A discharge electrode for use in the electrical purification of gases containing corrosive material is composed of a fused mass of glass, porcelain, quartz, white ware, or similar material together with electrically conducting bodies, *e.g.* metals. Alternatively a non-conducting core may be coated with this fused mass. J. S. G. THOMAS.

Plant for electrical precipitation. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 425,274, 15.9.23).—Discharge electrodes of U-cross-section are arranged above the upper ends of tubular precipitating electrodes which are inclined to the horizontal, and are so shaped that the discharge is effective only in the direction of apertures provided in the upper sides of the tubes, whilst a pure electrostatic field is produced in the opposite direction. The precipitating electrodes are provided with valves for discharging dust from the tubes to a collecting chamber containing gas for the combustion of the precipitated material. J. S. G. THOMAS.

Electrode for use in the electrolytic evolution of gases. E. HEINZE, Assr. to FARBENFABR. VORM. F. BAYER & CO. (U.S.P. 1,575,627, 9.3.26. Appl., 10.12.24).—An electrolytic cell for the production of gases contains a pair of electrodes in gas-tight spaced relation, one of which has within it a gas-collecting chamber and passages leading thereto from the space between the electrodes. M. E. NOTTAGE.

Purification of filter diaphragms in electrolytic processes. SIEMENS & HALSKE, Assees. of A. WEYL (G.P. 425,275, 3.12.24).—At intervals of time a cleansing solution comprising a solution of an electrolyte and an acid or alkaline solution is introduced in the neighbourhood of the diaphragm, the current strength being suitably reduced and the strengths of the acid or alkaline solution added so adjusted that the alkali or acid concentrations resulting from electrolysis at the cathode or anode are neutralised, or the solutions in these regions remain slightly acid or alkaline. J. S. G. THOMAS.

Filament [for electric lamps] and method of manufacture thereof. G. R. FONDA, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,565,724, 15.12.25. Appl., 29.7.20).—See E.P. 182,699; B., 1922, 673 A.

Coating [electric] incandescent lamp bulbs. J. B. WHITMORE and J. E. FERGUSON, Assrs. to WESTINGHOUSE LAMP CO. (U.S.P. 1,581,766, 20.4.26. Appl., 19.3.21).—See E.P. 230,643; B., 1925, 402.

Quartz [mercury vapour] lamp. K. MENSING (U.S.P. 1,582,849, 27.4.26. Appl., 24.8.21).—See E.P. 165,081; B., 1922, 742 A.

Production of compressed gases by electrolysis. P. Hausmeister (U.S.P. 1,581,944, 20.4.26. Appl., 17.11.23).—See E.P. 228,231 ; B., 1925, 249.

[Self-baking] electrodes for electric furnaces. B. E. D. Kilburn. From Det Norske Aktieselskab for Elektrokem. Ind. (E.P. 235,894, 5.1.25).

Emulsified solid greases (E.P. 232,259).—See II.

Lead amalgam (U.S.P. 1,570,763).—See X.

XII.—FATS; OILS; WAXES.

Identification of rapeseed oil by isolation of erucic acid. A. W. Thomas and M. Mattikow (J. Amer. Chem. Soc., 1926, 48, 968—981).—The oil (10 g.) is hydrolysed by a mixture of 50 c.c. of alcoholic 5% potassium hydroxide and 50 c.c. of 95% alcohol, the warm soap solution made neutral to phenolphthalein, and alcoholic potassium hydroxide added to restore a permanent pink colour. 25 c.c. of alcoholic magnesium acetate solution (50 g. dissolved in 100 c.c. of water and the solution diluted with 3 vols. of 95% alcohol) are added, the mixture is boiled, then kept at 10° overnight. The insoluble soaps are filtered off and washed with 50 c.c. of 95% alcohol, then washed back into the original flask with hot 5N-hydrochloric acid, decomposed by boiling for 10 min., and cooled. The solidified acids are filtered off, washed free from chloride and magnesium, transferred to a 150 c.c. beaker by means of warm 90% alcohol, and kept at 10° overnight. Crystals of saturated acid are then filtered off, and the filtrate is collected in a weighed 150 c.c. beaker. The solvent is slowly evaporated at 60—70°, drying being completed at 60° under reduced pressure. The iodine value, m.p., and mol. wt. of the acid are then determined. Pure rapeseed oil gives in this way about 44% of erucic acid, m.p. about 26°, iodine value about 73, mol. wt. 336. A confirmatory test consists in dissolving the acid in 80—100 c.c. of 90% alcohol, adding 2 c.c. of 1% palladous chloride solution and 0·5 c.c. of 1% gum arabic solution, and bubbling hydrogen through the mixture for about 3 hrs. The mixture is kept at 25° overnight, the precipitate collected, washed with cold 90% alcohol, and dissolved in hot 95% alcohol into a weighed beaker, in which the solvent is evaporated and the product dried at 80°. Pure rapeseed oil gives in this way about 33% of behenic acid, m.p. 77—79° after two recrystallisations. No "erucic acid product" is obtained by the above method from pure olive, cottonseed, linseed, soya bean, perilla, maize, wild mustard, or peanut (arachis) oils. The amount of erucic acid obtained from mixtures of rapeseed and olive oils gives a rough indication of the proportions of the two oils in the sample. Results obtained with mixtures of linseed and rapeseed oils indicate that the presence of large proportions of unsaturated acids interferes with the precipitation of magnesium erucate. Since erucic acid is a solid acid of low m.p., the "solid acids" of an oil will, in presence of rapeseed oil, show lower m.p., and data are given for the m.p. of the solid acids obtained from mixtures of olive and cottonseed oils with rapeseed oil. The above method does not distinguish rapeseed from mustard seed oil, as the latter yields a large proportion of erucic acid. F. G. Willson.

Method of Bertram, Bos, and Verhagen for the determination of coconut oil and milk fat. C. Baumann, J. Kuhlmann, and J. Grossfeld (Z. Unters. Lebensm., 1926, 51, 27—31 ; cf. B., 1926, 165).—The "A" and "B" numbers of some commercial milk-products and mixtures of coconut oil, milk fat, and cacao butter have been determined. While the method is less simple than the Reichert-Meissl or Polenske methods, it affords a clearer insight into the composition of the fats and is especially valuable for the determination of coconut oil in cacao butter. In the examination of milk products it is useful in detecting substitution of skim-milk powder for whole-milk powder or cream. The effects of variation in the conditions of experiment are discussed and the original method has been considerably simplified. E. H. Sharples.

Distillation of coconut oil at very low pressures. H. I. Waterman and H. J. Rijks (Z. Deuts. Oel- u. Fett-Ind., 1926, 46, 177—178).—The distillation is carried out in a glass flask, high vacuum being maintained by a Langmuir mercury pump as an auxiliary to an ordinary pump. Between the two pumps were two flasks containing charcoal cooled in liquid air. Between the ordinary receiver and the pump was an auxiliary receiver cooled in liquid air, followed by a vessel containing phosphorus pentoxide. Distillation temperatures were measured by thermo-elements. A vacuum of 0·0023—0·0034 mm. was maintained, as recorded by a MacLeod manometer. During distillation no ordinary ebullition was observed. Four fractions were collected, viz., 208—218°, 218—223°, 223—232°, and 232—259°, and amounted to 96·5% of the total. The refractive index, acidity, saponification value, and iodine value of the original material, calculated from that of the fractions, agreed closely with the actual observed figures, indicating absence of decomposition. It is suggested that by such methods of distillation it may be possible to separate the individual glycerides in fats. A. Rayner.

Determination of the bromine value of fats. E. Rupp and W. Brachmann (Z. anal. Chem., 1926, 68, 155—160).—To reduce the loss of bromine by evaporation in Winkler's bromide–bromate method of determining the bromine value of fats (B., 1924, 755) a large excess of potassium bromide should be present in the solution. A suitable standard solution consists of 5·5674 g. of potassium bromate dissolved in 1 litre of 20% potassium bromide solution. The excess of bromine is preferably determined by adding 0·3—0·5 g. of potassium iodide and titrating the liberated iodine with thiosulphate. Substitution of a 0·1N-solution of bromine in carbon tetrachloride or chloroform containing hydrogen chloride for the aqueous bromide–bromate mixture yields low results, probably because water acts as a catalyst in the latter case. A. R. Powell.

Theory of the practice of steam deodorisation of saponifiable oils. W. BRASH (J.S.C.I., 1926, 45, 73—75 T).—The object of steam deodorisation of saponifiable oils is the removal of those substances giving rise to taste and odour; they are neutral compounds and in coconut and palm kernel oils have been found to consist largely of the higher aliphatic ketones. The theory of the process is developed and, with certain assumptions, it is shown that the concentration, x, of the odoriferous matter in the oil after deodorisation for a time, t, is given by the equation $x=a\,e^{-Kkt}$ where a is the original concentration of the odoriferous matter, and K and k are constants. Formulæ are given for the design of suitable apparatus and for calculating the pressure and temperature of the entering steam.

Bleaching of wool grease. I. LIFSCHÜTZ (Chem.-Ztg., 1926, 50, 245—246).—The bleaching of wool grease on exposure to air and light is due to oxidation of the products of hydrolysis (alcohols and fatty acids) of the esters, the undecomposed esters not being bleached. This principle has been applied on a large scale in conjunction with the process described in G.P. 324,667 (B., 1920, 790 A) for bleaching wool grease. The sodium soap of the completely saponified grease, pressed into thin flakes, was exposed to sunlight in glass boxes. After 3—4 days the dark brown soap flakes had become bleached to a pale yellow. If the grease was only partly saponified with lime the results were much inferior. The process is stated to be applicable on a large scale to all grades of wool grease, and the sodium soaps may be used for many industrial purposes. They are particularly suitable as additions to toilet soaps, as in virtue of the nature of the unsaponifiable matter present the lathering and detergent properties of the soap are improved. A. RAYNER.

Polymerisation of fatty oils. V. J. MARCUSSON (Z. angew. Chem., 1926, 476—478; cf. B., 1925, 889).—Linseed oil films contain about 55% of solid polymerised and oxidised gel and 40% of polymerised and oxidised sol with 5% of unchanged oil. Poppy-seed oil films contain 51% of insoluble gel (mol. wt. 707), 31% of easily gelatinised sol containing 40% of unpolymerised fatty acids and a variable amount of acids of mol. wt. 420, and 18% of unchanged oil. The acetone-insoluble gel of tung oil films (83%) contains a unimolecular, oily product soluble in furfuraldehyde and having a mol. wt. of over 100 by Rast's camphor method. During the drying of tung oil, isomerisation due to the action of light first takes place, followed by polymerisation and oxidation. The halogens and sulphur have a similar action to light in that at 10° they precipitate β-elæostearin and at room temperature a solid polymerisation product. For the detection of tung oil in mixtures 5 g. of the oil are shaken with 5 c.c. of a cold saturated solution of iodine in chloroform; with high percentages of tung oil a gelatinous mass is obtained but with 15—20% of tung oil the mixture must be warmed on the water-bath. No other fatty oil behaves similarly. A rough quantitative test may be made by extracting the reaction product with chloroform in Graefe's apparatus, removing the iodine by shaking with thiosulphate, distilling off the chloroform, drying, and weighing. A. R. POWELL.

See also A., May, 471, **Viscosity and elasticity of soap solutions** (FREUNDLICH and JORES). 481, **Partial saponification of mixed glycerides** (TREUB). 498, **Chinese wood (tung) oil** (NAGEL and GRÜSS).

Vitamin-A in oleo oil and oleo stearin. HOAGLAND and SNIDER.—See XIX.

PATENTS.

Simultaneous purification and deacidification of oils and fats. KUNEROLWERKE E. KHUNER & SOHN A.-G. (Austr. P. 96,831, 13.11.22).—The oil or fat is treated with oxidising agents having a neutralising action, especially sodium peroxide, which can be used in solution. S. S. WOOLF.

Emulsified solid greases (E.P. 232,259).—See II.

Gilsonitic products (U.S.P. 1,573,764—5 and 1,578,235).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Chrome yellow problems. H. WAGNER and E. KEIDEL (Farben-Ztg., 1926, 31, 1567—1573).—Many precipitations of pure lead chromate and of various mixtures of lead sulphate and chromate were carried out with variations in the temperature, concentration, agitation, precipitants used, etc. By sedimentation experiments and microscopical examination the state of dispersion of the product was shown to be dependent on its solubility in the mother liquor. This in turn is controlled by the salts used and the practical conditions in general, concentration being the most important variable. In the case of chrome yellow containing lead sulphate the existence of mixed crystals was recognised and their bearing on the tone, opacity, etc. of the pigment is discussed. The optimum conditions deduced from this investigation agree with those empirically found to be practical for any desired type of chrome yellow. S. S. WOOLF.

Photometric method for measuring the hiding power of [light-coloured] paints. H. D. BRUCE (U.S. Bur. Standards Tech. Papers, 1926, 20, [306], 173—190).—The paint under test is poured on to a rotating circular glass disc—half black and half white—and allowed to dry, producing a dried film tapering from a slight peak in the centre to a minimum at the outer edges. The relative brightness ("contrast ratio") of the two halves of the plate for different thicknesses of the covering film is measured by a Martens photometer (in which the two fields are brought to equal brightness by means of a Nicol prism; cf. Physikal. Z., 1900, 1, 299). Film thickness is obtained by the use of an Ames dial, a reading being taken on the film, which is then removed with a penknife and a second reading taken

on the bare plate. The minimum thickness of film (x) that will hide "completely" (except for personal equation) is computed from the formula $x=7a\sqrt{1/b}-1$, where a is the measured film thickness and b the measured contrast ratio. This formula is a simplified form of the functional relationship developed in the paper. In addition, the hiding power may readily be expressed in square feet per gallon. S. S. WOOLF.

Adsorption of soluble salts by paint films. H. WOLFF and G. ZEIDLER (Korrosion u. Metallschutz, 1925, **1**, 211—213; Chem. Zentr., 1926, I., 2746).—Sodium chloride can readily be dissolved out of films of oil paints containing, in addition to the salt, zinc oxide, lithopone, and red oxide of iron respectively, but corresponding films containing white lead or iron–mica retain the salt to a considerable extent. The interfacial tension relationships in the system pigment, salt, oil, and water must be such as to permit adsorption in the last two cases. If concentrated sodium chloride solution be stirred into the paint, instead of incorporating the dry salt, the washing out proceeds more rapidly owing to water retained in the film assisting diffusion. Rain cannot completely wash salt out of iron–mica films. S. S. WOOLF.

Natural and artificial resins. J. SCHEIBER (Farbe u. Lack, 1926, 76—78, 87—89, 99—101, 113—114).—An account of the published work on various types of resins, natural and synthetic, in use in the paint and varnish as well as other industries, mainly from theoretical and constitutional standpoints. Under the heading natural resins, Tschirch's general classification, rosin, copals, resin–oil interactions, shellac, and Japanese lacquer receive attention, while under artificial products a short account of resin formation and "resinophore groups" is followed by discussion of polymerides of coumarone, indene, and aldehydes, and condensation products of phenol or carbamide with formaldehyde and the many similar patented products. S. S. WOOLF.

Frosting of tung oil films. The tung oil phenomenon. L. AUER (Farben-Ztg., 1926, **31**, 1625—1627).—At least three theories have been advanced to explain the characteristic frosted appearance of tung oil films dried under ordinary atmospheric conditions. According to Marcusson and others, the separation under the influence of light of crystals of β-elæostearin, the solid isomeride of α-elæostearin, which comprises most of the original film, is responsible for the opacity of the dried film, but macro- and microscopical examinations (as well as analyses by recent workers) show the absence of these crystals and establish wrinkling as the cause. When the puckers are sufficiently fine, diffraction and iridescence occur. Wolff, Schmidt, and others attribute this wrinkling to internal strain of the colloid system, set up by swelling and shrinking with absorption and disengaging of moisture, as evidenced by the possibility of obtaining clear films in dry atmospheres. The author now shows that non-frosted films may be obtained in desiccators where water exchanges are proved to be proceeding, and affirms that ultra-violet light is the deciding, if not the only, cause of the frosting. The glass of which the desiccators used are made absorbs ultra-violet light almost completely, but clear films taken out of the desiccator at a not too advanced stage in the drying and exposed to sunlight, rapidly develop the usual markings. It is denied that frosted films can be obtained in the complete absence of light as has been claimed by some workers. S. S. WOOLF.

Ester gum [rosin glyceride] and the chemical reactions in rosinate varnish preparation. H. WOLFF (Farben-Ztg., 1926, **31**, 1573—1574).—The slight sediment in a 50% amyl acetate solution of commercial ester gum after keeping for 3 months, was found on recrystallisation etc. to be pure glyceryl triabietate. By using these crystals to seed an ester gum solution in ethyl acetate (33%) further quantities were obtained. This separation throws light on the exchanges between fatty acid glycerides and metal rosinates in varnishes, as a sample of triabietin, identical with the above, was isolated from a linseed oil solution of lead rosinate, thus confirming the earlier views of Wolff and Dorn (cf. B., 1921, 780 A, 857 A). The saponification value and molecular weight etc. of a secondary product obtained from the triabietin mother liquor agree with the values for an impure abietic anhydride. This, it is suggested, is derived from the lead rosinate by the splitting off of litharge which then by saponifying the oil glycerides frees glycerol residues to form triabietin with the abietic anhydride. S. S. WOOLF.

U.S. Government master specification for shellac varnish. (U.S. Bur. Standards Circ. 303, Feb. 9, 1926, 7 pp.).—Two types each of orange and bleached shellac varnish are specified, and each type may be provided at light, medium, or heavy body. The requirements for the non-volatile matter in the varnishes are as follows:—Maximum iodine value for orange, type I, 18·0, type II, 24·5, bleached, types I and II, 10·0. Maximum percentage of insoluble matter in hot 95% alcohol, orange type I, 1·75, type II, 3·00, bleached type I, 1·00, type II, 0·10. Maximum percentage of wax, orange types I and II and bleached type I, 5·50, bleached type II, 0·20. Maximum ash, orange types I and II and bleached type I, 1·00, bleached type II, 0·30. The colour should be no darker than that of samples mutually agreed on by buyer and seller. The body of the varnishes is controlled by minimum specified percentages of non-volatile matter, viz: for orange types I and II, light 33·4, medium 36·4, and heavy 39·1. For bleached types I and II, light 32·3, medium 35·3, and heavy 37·9. Details of reagents and manner of testing are given. S. S. WOOLF.

Polymerisation of fatty oils. MARCUSSON.—See XII.

PATENTS.

Manufacture of a substance for impregnating materials. NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (E.P. 239,500, 15.8.25. Conv.

2.9.24).—The impregnating substance made by incorporating oil and sulphur with pitch, according to E.P. 154,570 (B., 1921, 885 A) is improved if bitumen produced from raw petroleum with an asphalt basis is the particular pitch substance used. A typical product is obtained by heating 100 pts. of castor oil with 30 pts. of sulphur for ½ hr. at 200°, and after adding 150 pts. of the petroleum pitch, maintaining the whole at 170° for 1 hr., stirring throughout. S. S. WOOLF.

Fluxed resinous composition. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,579,195, 30.3.26. Appl., 17.12.24).—Hexamethylenetetramine is added to resinous condensation products of the type ordinarily hardened by hexamethylenetetramine in the hot press to delay their too rapid setting and hardening in the mould, thus improving the finish of moulded articles. S. S. WOOLF.

Production of resinous products from crude anthracene and phenanthrene. BAKELITE G.M.B.H., and M. FLORENZ (G.P. 420,443, 30.12.22). —Anthracene and phenanthrene or their mixtures are converted into addition products and the latter resinified by heating, if necessary, with aldehydes, in the presence of condensing agents (sodium hydroxide, ammonia, ferric or aluminium chloride, etc.). The addition products are obtained by the action of halogens, picric acid, nitric acid or substances yielding nitrous fumes, such as nitrosyl chloride. For example, crude anthracene is suspended in carbon disulphide or benzene and saturated with chlorine, the medium evaporated at 100°, and the residue heated to melting above 100° till no more anthraquinone distils and the mass solidifies to a lustrous black resin. These resins, m.p. 80—90°, are soluble in benzene and halogenated hydrocarbons, and are used in the lacquer industry, for impregnating wood, and for insulation etc. B. FULLMAN.

Preparation of artificial resins. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of A. VOSS (G.P. 422,910, 30.7.21).—Natural resins are fused with synthetic resinous products containing free carboxyl groups. If oxides, hydroxides, or carbonates of the alkaline-earths are included in the melt, the products have much higher softening points than those obtained by fusion of natural resins or their salts with phenol-aldehyde condensation products. German spruce resin, on fusion with the resinous product from salicylic acid and formaldehyde, first at 100—120°, and then at 190—200°, gives a resin having a softening point at least 30° above that of spruce resin. Colophony and the condensation product of phenoxyacetic acid and formaldehyde yield a product suitable for lacquers and for electrotechnical uses. B. FULLMAN.

Distillation of natural resins and oleoresins. J. M. A. CHEVALIER, P. BOURCET, and H. REGNAULT (U.S.P. 1,572,766, 9.2.26. Appl., 17.5.22).—The resin or oleoresin is mixed with 1—5% of a tribasic acid, *e.g.*, phosphoric acid, and distilled to 310°. About 50% of the resin distils and is obtained in the form of a light oil useful as a substitute for turpentine and as a carburetting agent for alcohol. It contains a small proportion of phosphoric acid and water which is readily separated. The residue in the still consists of an odourless neutral resin oil of low viscosity. T. S. WHEELER.

Manufacture of [resinous] phenolic condensation products. H. WADE. From S. KARPEN AND BROS. (E.P. 245,703, 23.10.25).—See U.S.P. 1,566,817; B., 1926, 202.

Polyhydric alcohol–organic acid resinous condensation products. J. G. E. WRIGHT, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,581,902, 20.4.26. Appl., 5.7.24).—See E.P. 236,591; B., 1925, 770.

Manufacture of resinous products of condensation from phenol and formaldehyde. C. KULAS and C. PAULING (U.S.P. 1,582,056, 27.4.26. Appl., 13.8.21).—See E.P. 191,417; B., 1923, 235 A.

Production of hard resinous bodies. E. SCHAAL (U.S.P. 1,583,014, 4.5.26. Appl., 25.8.24).—See E.P. 243,556; B., 1926, 137.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Physical properties of rubber. I. Influence of high temperature on the stress–strain curve of vulcanised rubber. A. VAN ROSSEM and H. VAN DER MEYDEN (J.S.C.I., 1926, 45, 67—72 T).—Tensile tests at high temperatures were carried out with the dynamometer of Schopper by heating the pulleys supporting the rings in an oven at the temperature required and quickly mounting them. Subsequently the ring, which is heated under mercury at the temperature required, is quickly mounted and the tensile test is immediately carried out. Systematic experiments with vulcanised rubber (rubber 92½ pts., sulphur 7½ pts.) showed that with rising temperature the tensile curves shift towards the elongation axis. When the rings are heated for a longer time under mercury the rubber becomes brittle but the brittleness disappears after cooling at ordinary temperature. The tensile curve of the rubber after heating and subsequent cooling coincides with the original stress–strain curve of the rubber. The time of heating necessary for the appearance of brittleness depends largely on the vulcanisation coefficient and the temperature of heating. The change in tensile properties during vulcanisation has been studied by vulcanising rings in special moulds and immediately testing them on pulleys heated at 147°. The tensile properties of the rubber during vulcanisation are entirely different from those after cooling at ordinary temperature, the tensile strength being very low during vulcanisation and the rubber soon becoming brittle.

Commoner mineral ingredients for rubber. D. F. TWISS and E. A. MURPHY (J. S. C. I., 1926, 45, 121—123 T).—Progressive vulcanisation of mixtures of rubber and sulphur with various compounding ingredients shows that, apart from reinforcing effect, these may exert a distinctly divergent effect on the rate of vulcanisation.

Factice-like product obtained by the action of the silent discharge on oil ("Voltolfactice"). L. HOCK (Kautschuk, March, 1926, 65—67).—In the conversion of mobile oils such as rape oil into more viscous "voltol oils" by submission to the silent electric discharge in an atmosphere of hydrogen or other inert gas an insoluble deposit of "voltolfactice" is also obtained. This is a yellowish spongy mass of fatty odour, resembling white factice (oil rubber-substitute) in appearance. Its mixtures with rubber are inferior to similar mixtures with dark or light rubber substitute both in cold and hot vulcanisation, particularly the latter. D. F. TWISS.

Effect of accelerated ageing upon some physical properties of hard rubber compounds [vulcanites]. E. O. DIETERICH and H. GRAY (Ind. Eng. Chem., 1926, 18, 428—430).—Of the three physical characteristics, softening temperature, transverse strength, and impact strength, the last is the most promising as an index of the changes in vulcanite on ageing. At 70° the physical properties undergo little change even in 14 days, but at 150° deterioration is rapid. The use of vulcanisation accelerators or age-retarders has little influence, but under-vulcanisation favours deterioration. Heavily loaded stocks show a lower percentage alteration than simple vulcanites prepared from rubber, sulphur, and an accelerator. D. F. TWISS.

Chemical unsaturation of rubber under the action of heat, trichloroacetic acid, ultra-violet light, and mastication. H. L. FISHER and A. E. GRAY (Ind. Eng. Chem., 1926, 18, 414—416; cf. Staudinger, B., 1926, 282).—At 340° rubber yields a powdery product of a much lower degree of unsaturation; at 141°, however, even after 8 hrs. there is no decrease in the unsaturation towards Wijs' solution. Rubber, in which 10% of trichloroacetic acid has been incorporated, shows a fall in its unsaturation value after 7 days. Ultra-violet light does not cause any alteration in the unsaturation of rubber solution in an atmosphere of carbon dioxide. Mastication of rubber lowers the degree of unsaturation probably on account of oxidation; with exclusion of air the unsaturation is not affected. D. F. TWISS.

Dielectric constant, power factor and resistivity of rubber and gutta-percha. H. L. CURTIS and A. T. MCPHERSON (U.S. Bur. Standards Tech. Papers, 1925, 19, [299], 699—722).—Well-washed rubber has a dielectric constant (2·35) lower than that of the purest gutta-percha examined (2·56), and the values are more variable for gutta-percha. Vulcanisation increases the dielectric constant and the power factor of rubber, but the use of accelerators results in lower values than are obtained on vulcanisation with sulphur alone; the resistivity is little affected by vulcanisation. Softeners such as ozokerite, vaseline, beeswax, palm oil, and stearic acid have little influence on the dielectric constant of rubber, but coumarone resin and large proportions of "mineral rubber" cause a slight increase; with the exception of ozokerite, they all increase the power factor considerably. Mineral fillers generally increase the dielectric constant of rubber, the increase, however, being relatively slow with increasing proportions of quartz powder, which is also exceptional in actually reducing the power factor; the resistivity is not greatly influenced by moderate proportions of most fillers. Carbon black, however, is exceptional in the degree of its effect in increasing the dielectric constant and power factor and reducing the resistivity. Absorption of water increases the dielectric constant of rubber (and gutta-percha) and the power loss and decreases the resistivity; latex-sprayed rubber, on account of its content of soluble matter, exhibits a much higher water absorption than pale crêpe rubber. D. F. TWISS.

PATENT.

Impregnation of wood (Austr. P., 100,729).—See IX.

XV.—LEATHER; GLUE.

Possible explanation of the antagonistic action of neutral salts upon hide substance. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1926, 21, 206—216).—The explanation is based on the secondary valency concept. Where the destructive action of neutral salts upon hide substance prevails, there is a tendency to the formation of neutral salt compounds similar to the glycine–calcium chloride compounds described by Pfeiffer ("Org. Molekülverbindungen, Stuttgart," 106—124). The extent of this peptisation depends upon the magnitude and stability of the secondary valency between the salt and protein micelles. A weakening and partial breaking up of the intermolecular forces occur by this combination and part of the hide is transformed into micelles or complexes of molecular disperse order. D. WOODROFFE.

Action of chromium salts on permutit. Indirect proof of the chemical nature of one-bath chrome tanning. K. H. GUSTAVSON (Collegium, 1926, 97—124).—The behaviour of permutit with different chromium salt solutions is shown to resemble that of collagen, *e.g.*, in regard to effect of temperature, of concentration, and of basicity. The base exchange with chromium chloride solutions is, however, not the same for both permutit and hide substance. The action of chromium chloride solutions of all degrees of basicity on permutit indicates the presence of a tervalent cation. The basic chromium sulphates react with permutit as if there were complex chromium cations of less than three valencies. The maximum base exchange takes place in liquors of high basicity. Diminution of basicity or dilution results in a gradual increase in the valency. Basic chromium sulphates differ from the chlorides in showing greater tendencies to polymerisation and condensation apparently owing to the residual affinities of the hydroxyl groups. The diffusibility of the basic chromium sulphates depends on the basicity, whereas that of the basic chromium chlorides is independent of the basicity. The basicity of the cation in chromium chloride is

about 90%. This agrees with its physico-chemical nature, the basicity at which this salt precipitates, and its inferior tanning properties. Addition of up to 2 mols. of sodium chloride to basic chromium chloride solutions of 54·8% basicity caused an increase in the amount of chromium fixed by the permutit and collagen and a lowering of the p_H figure and of the basicity at which precipitation commenced. The neutral salt effect cannot be explained by the hydration hypothesis alone; there are constitutional changes also. The hydrion concentration is not the only determining factor in the absorption of chromium, which is affected also by the nature of the cation, the number of valencies, the properties of the acid and basic groups, and also the degree of aggregation. Very small additions of sodium sulphate lower the precipitation figure and the hydrion concentration. The absorption of chromium compounds from such solutions by collagen and permutit agrees with the view that the tendency to form chromium complexes is greater with sulphates than with chlorides. The valency of the cations is diminished and polymerisation and condensation processes are initiated. Increasing amounts of sodium sulphate cause increased formation of negatively charged chromium complexes, which can combine with collagen but not with permutit. The addition of small quantities of sodium sulphate to chromium chloride solutions causes an appreciable increase in their tanning power. Chrome tanning is due partly to the ordinary valencies and partly to residual valencies of the chromium compounds.

D. WOODROFFE.

Quinone tannage. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1926, 18, 383—385; cf. B., 1924, 918).—The addition of sodium chloride retards the rate of quinone tanning in alkaline solutions. In neutral solutions, sodium sulphate aids the tanning and sodium chloride retards it. The amounts of "tannin" removed by alcohol were compared with the p_H values of the tanning solutions and it was found that alkaline-tanned leathers were more resistant to alcohol treatment the higher the p_H value of the tanning solution. The percentages of material extracted by alcohol determined from the weight of extract and from loss in weight of the leather showed that as fixed "tannin" is removed, something else is fixed by the leather. It is suggested that this is acetaldehyde. The same phenomenon has been observed with leathers tanned with hemlock bark and gambier. Very little gallotannic acid is fixed by quinone-tanned leather. The aqueous extraction of quinone-tanned leather at different p_H values resulted in a gradual loss in weight of the leather in alkaline solutions. Quinone tannage appears to take place at the same groups of the collagen molecule as vegetable tannage.

D. WOODROFFE.

Properties of shoe leather. I. Micro-structure J. A. WILSON and G. DAUB. **II. Chemical composition.** J. A. WILSON and S. O. LINEAR (J. Amer. Leather Chem. Assoc., 1926, 21, 193—198, 198—206). —I. Photomicrographs of sections of representative samples of 18 varieties of shoe leather are reproduced. Sections of vegetable-tanned and chrome-tanned calf-skins respectively show that in the vegetable tannage the fibres are built up to a much greater extent than in the chrome tannage, which makes the vegetable-tanned leather relatively tight and full compared to the looser and more open structure of the chrome leather. The sections of the various kinds of leather show characteristic differences of structure. II. The complete chemical analyses of 18 leathers mentioned above are given in the following table, viz.: (1) coloured vegetable-tanned calf, (2) coloured chrome-tanned calf, (3) black chrome-tanned glazed kid, (4) black chrome-tanned kangaroo, (5) vegetable-tanned horse bull (cordovan), (6) chrome-tanned buffed and split cow hide, (7) chrome-tanned split cow hide, (8) black chrome-tanned "slink" calf (suede), (9) russet lining calf, (10) russet lining sheep, (11) vegetable-tanned shark, (12) patent chrome split cow hide, (13) patent chrome-tanned kid, (14) patent chrome colt, (15) heavy chrome-tanned cow hide, (16) chrome re-tan army upper leather, (17) vegetable-tanned sole eather, (18) chrome sole leather.

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Water	13·6	16·3	13·7	12·0	10·0	14·1	16·3	12·7	11·9	10·9	12·2	10·1	11·8	12·0	14·4	15·1	14·6	16·3
Skin protein (N × 5·62).. ..	41·0	62·6	65·3	62·7	40·1	69·6	66·8	55·1	46·0	50·0	45·4	50·5	54·0	60·4	57·0	44·6	29·7	29·4
Fat	12·0	4·6	6·6	11·3	18·6	2·1	5·8	7·1	7·6	6·1	6·9	10·0	6·6	5·1	14·2	20·4	3·2	25·4
H_2SO_4	0·3	3·4	1·0	1·8	0·6	3·2	3·6	0·8	0·1	1·7	1·5	1·8	2·1	2·3	4·4	1·1	0·8	5·9
Al_2O_3	0·4	1·2	0·2	0·1	—	1·0	0·2	1·0	0·1	0·1	—	0·1	0·2	0·1	—	0·3	—	2·6
Fe_2O_3	0·1	0·3	0·3	0·1	0·1	0·6	0·2	1·2	—	0·1	0·1	0·4	0·6	0·3	0·7	0·2	0·7	0·5
Na_2SO_4	—	0·4	0·9	0·2	—	0·3	1·0	0·4	—	—	—	0·6	0·3	0·5	0·4	0·3	—	12·3
HCl	—	0·3	0·5	0·3	—	—	—	0·2	—	0·6	—	0·1	—	0·1	0·1	—	—	0·8
NaCl	—	0·5	0·2	0·1	—	—	0·3	0·1	—	—	—	—	—	0·1	0·4	0·4	—	0·9
Cr_2O_3	—	5·4	4·5	6·0	—	5·3	3·6	5·4	—	—	—	2·9	3·6	3·6	5·5	2·4	—	1·7
CaO	—	—	0·2	0·3	0·1	0·2	0·1	0·2	0·2	0·1	0·1	0·1	0·2	0·3	—	—	—	—
$MgSO_4$	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	0·8	0·5
$CaSO_4$	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	2·3
Collodion	—	—	—	—	—	—	—	—	—	—	—	9·0	8·4	6·1	—	—	—	—
Soluble organic matter ..	9·1	—	—	—	8·7	—	—	—	12·3	13·0	5·4	—	—	—	—	—	35·6	—
Combined tannin (by diff.) ..	23·5	—	—	—	21·8	—	—	—	21·8	17·4	28·4	—	—	—	—	—	14·6	—
Other organic matter (by diff.)..	—	5·0	·6	5·16	—	3·6	2·11	5·8	—	—	—	14·4	12·2	9·1	2·9	15·2	—	1·4

The fat was extracted with choroform. After extraction with chloroform patent leathers were extracted further with a mixture of ethyl acetate and acetone for several days. The residue from this further extract was called collodion. In the chrome leather analyses, H_2SO_4 represents the combined acid sulphate determined according to the official

methods of the American Leather Chemists Association. Water-soluble matter was determined by extracting the fat-free leather with water in a Wilson–Kern extractor until the wash water gave no coloration with ferric chloride solution. D. Woodroffe.

Patents.

Depilation of hides and skins. F. Ullman, Assee. of H. Benfey (E.P. 246,114, 17.12.25. Conv., 13.1.25).—Solutions for the depilation of hides and skins contain lime and less than 1% of sodium hydrosulphide. L. A. Coles.

Tanning composition. Badische Anilin- & Soda-Fabr., Assees. of C. Immerheiser and H. Wolff (U.S.P. 1,557,844, 20.10.25. Appl., 9.7.20).—See E.P. 146,427 and 169,943; B., 1921, 819 a, 820 a.

Tanning and the manufacture of tanning materials. J. A. S. Morrison (U.S.P. 1,581,224, 20.4.26. Appl., 23.1.24).—See E.P. 219,347; B., 1924, 841.

XVI.—AGRICULTURE.

Spontaneous multiplication of [nitrogen] fixing organisms [in soil]. S. Winogradsky (Compt. rend., 1926, **182**, 999—1001).—Addition of 0·5—1% of dextrose or mannitol to a fertile soil kept damp at 30°, results in an enormous increase in the number of *Azotobacter* present. Experiments in Petri dishes on a starch medium give almost completely pure colonies. These facts may be utilised in classifying soils according to their nitrogen-fixing capacity. Other substances which increase the C : N ratio in the soil, *e.g.*, other carbohydrates, alcohols, and organic acids, have a similar, but generally less effect. Increase of nitric acid or ammonia in the soil, on the other hand, results in a decrease in the number of *Azotobacter* and an increase of bacteria which are harmful to the soil. When 0·005% of nitric acid was added to a soil 96% of the *Azotobacter* disappeared; when 0·01% of nitric acid was added multiplication of *Azotobacter* ceased and the soil was invaded by other organisms. L. F. Hewitt.

Effect of basic slag on the lime status of soils. R. Williams (J. Agric. Sci., 1926, **16**, 196—204).—In laboratory experiments, addition of 0·1% of basic slag to soils with a low percentage of exchangeable calcium and a low degree of saturation causes an increase in the exchangeable calcium, the degree of saturation, and the calcium soluble in an aqueous solution of carbon dioxide, similar to that obtained on adding equivalent amounts of calcium oxide or carbonate. The effect on p_H is less marked. The soil of field plots treated with basic slag also showed an increase in exchangeable calcium which was still evident 8 years after the application of the slag. C. T. Gimingham.

Rôle of phosphorus in agriculture. E. Vanstone (J.S.C.I., 1926, **45**, 78—80t).—The importance of an adequate supply of phosphorus and lime in maintaining soil fertility is discussed and illustrated by results of experiments at home and abroad. A brief account of some of the author's work on soil phosphates and phosphatic fertilisers is given with special reference to methods of determining the availability of the phosphorus.

Standard methods of analysis of fertilisers. Edited by J. Sen (Bull. Agric. Res. Inst. Pusa, 1926, [164], 14 pp.).—Details are published of standard procedures for the analysis of fertilisers, largely based on the methods of the Association of Official Agricultural Chemists of the U.S.A. These include determinations of moisture, total, water-soluble, and citrate-soluble P_2O_5, total, nitric, and ammoniacal nitrogen, and K_2O by both the platinum and perchlorate methods. C. T. Gimingham.

Detection of leather in fertilisers made from waste products. G. Joret and E. Radet (Ann. Falsif., 1926, **19**, 148—152).—If leather has been used in the manufacture of the fertiliser tannin can usually be detected in the boiling water or oxalic acid extracts. As a rule the darker and more acid the sample the more readily is tannin to be detected, and the larger the proportion of calcium phosphate added to the fertiliser the more difficult does the detection become. If a negative result is obtained with the oxalic acid extract the product should be warmed with successive small portions of hydrochloric acid till an excess of acid is present; a little warm water is then added, the material filtered, and the insoluble residue washed several times, and then treated with boiling oxalic acid when, if leather was originally present, a tannin reaction will be obtained. D. G. Hewer.

Determination of ammoniacal nitrogen in fertilisers. F. Chastellain (Helv. Chim. Acta, 1926, **9**, 205—216).—The Ronchèse method (A., 1907, ii, 651) of determining ammonia by treatment with formaldehyde and titration of the acid liberated can be applied to fertilisers, using phenolphthalein as indicator. Where hydrolytic interference occurs in the presence of compounds such as dicyanodiamidine salts, phenolphthalein is replaced by rosolic acid but phosphoric acid must be absent. For fertilisers of the superphosphate type, an aqueous extract is treated with milk of lime and sodium carbonate and the ammonia in an aliquot portion of the filtrate is determined by the Ronchèse method using rosolic acid. Ammonia is removed completely from solutions of its salts by boiling with calcined magnesia and since little decomposition of urea and dicyanodiamidine is caused by this process, it can be applied to the determination of ammonia in fertilisers. When ammonia-free air is bubbled through two cold solutions containing known amounts of ammonium salts and sodium hydroxide, the ratio of the ammonia removed from each at any given time is constant and is the same as the ratio of the ammonia in the solutions. Since sodium hydroxide solutions containing up to 24% of alkali have little action on urea and dicyanodiamidine, the ammonia in fertilisers can be determined by passing a stream of air through a flask containing a standard amount

of ammonium sulphate, into which excess of sodium hydroxide is run, the ammonia carried over being collected in a known volume of dilute sulphuric acid; the air stream is then passed through a solution of the fertiliser and alkali and finally through another standard sulphuric acid solution. At the end of two hours, the amounts of ammonia carried over are determined. If m be the standard weight of ammonia, m_1 and m_2 the weights of ammonia carried over, then the weight of ammonia in the fertiliser is $m, \times m_2/m_1$. This method is recommended for general use in determination of ammonia in calcium cyanamide fertilisers. M. CLARK.

Salt requirements of *Lupinus albus*. C. H. ARNDT (Soil Sci., 1926, **21**, 1—6).—Seedlings of *Lupinus albus* were grown in culture solutions containing varying ratios of nutrient salts. The optimum salt ratios were 5:3:4 for potassium, calcium, and magnesium, and 5:9:4 for nitrate, phosphate, and sulphate. A favourable total salt concentration was found to be 0·0084N. Excess of phosphate in the culture solutions tended to produce chlorosis. No evidence was obtained to justify the term "calciphobe" as applied by geologists to *Lupinus albus*. A. G. POLLARD.

Remarkable correlation between [yields of] grain and straw. W. A. MACKENZIE (J. Agric. Sci., 1926, **16**, 275—279).—A set of 46 unmanured plots (each 0·098 acre) was sown with wheat to test the uniformity of the soil of a field at Rothamsted. The soil was found to be very variable in fertility, but an exceedingly close association between the yields of grain and straw on the plots was evident. The correlation coefficient calculated from the 46 pairs of values was +0·990. C. T. GIMINGHAM.

Nutritive value of pasture. I. Seasonal variations in the productivity, botanical and chemical composition, and nutritive value of medium pasturage on light sandy soil. H. E. WOODMAN, D. L. BLUNT, and J. STEWART (J. Agric. Sci., 1926, **16**, 205—274).—A detailed study of the nutritive value throughout the season of the herbage of a pasture on light soil under grazing conditions. The influence of meteorological conditions on the yield is discussed; there was a marked correlation between yield and rainfall during a period of low rainfall in June and July. The total yield per acre from hay plots was more than twice as great as that from the pasture, but, in comparison with the hay, there was a very high percentage of protein and a low percentage of fibre in the pasture grass. The digestion trials showed that well grazed pasture possesses a much higher nutritive value than has hitherto been thought, and compares with concentrates like linseed cake. The high degree of digestibility of the fibre is specially notable. The digestibility of the grass varied during the season, but, at its lowest, was superior to that of the best meadow hay. The pasture plot produced considerably more digestible protein than the hay plot. Seasonal changes in the lime and phosphate content of the pasture grass and the utilisation of these constituents by the sheep were also followed and are considered in detail. The lime content of the grass increased to a maximum in the dry period and declined later. The phosphate behaved in the opposite manner. The system of frequent cutting favoured the spread of wild white clover on the plot to a remarkable extent. Suggestions are made with regard to the management of pasture and meadow, based on the results obtained. C. T. GIMINGHAM.

Rôle of calcium hydroxide in hydrated lime-acid lead arsenate sprays. F. L. CAMPBELL (J. Agric. Res., 1926, **32**, 77—82).—The success of hydrated lime in reducing the injury to foliage caused by acid lead arsenate alone may be due to the fact that soluble calcium arsenate is less toxic than soluble arsenic acid derived by hydrolysis of the acid lead arsenate. In humid conditions, however, acid calcium arsenate may be formed in sufficient concentration to cause foliage injury. H. J. CHANNON.

Fungicidal properties of certain spray fluids. IV. W. GOODWIN, H. MARTIN, and E. S. SALMON (J. Agric. Sci., 1926, **16**, 302—317; cf. Goodwin and Martin, B., 1925, 780).—The toxicity of various substances to the conidal stage of the hop mildew (*Sphaerotheca humuli*) has been compared under standardised conditions. Solutions of potassium, sodium, and calcium polysulphides proved fungicidal at approximately the same concentration of polysulphide sulphur (0·092 to 0·12%). The fungicidal concentration of dicalcium and disodium arsenates is 0·024% As_2O_5; of lead thioarsenate, 0·05%; and of lead arsenate, between 0·1 and 0·2%. A mixture of calcium polysulphide and lead arsenate is superior in fungicidal value to either of its components; solutions at concentrations below fungicidal strength, when mixed, proved to be fungicidal. This is probably accounted for by the formation of soluble arsenates and thioarsenates. C. T. GIMINGHAM.

Alternate extraction and exposure method for the study of arsenicals. S. LOMANITZ (J. Agric. Res., 1926, **32**, 499—500).—In order to determine water-soluble arsenic in arsenical insecticides under conditions approximating to those occurring in field practice, a method involving several successive extractions and exposures of the residue to the air is proposed. The results thus obtained give more reliable information as to the risk of foliage injury from the use of arsenicals than a single determination. C. T. GIMINGHAM.

Practicability of quantitative toxicological investigations on mandibulate insects. F. L. CAMPBELL (J. Agric. Res., 1926, **32**, 359—366).—A method for the quantitative study of the toxicity to insects of soluble poisons, taken through the mouth, is based on the observation that certain caterpillars will drink completely drops of liquid placed in their feeding path. The results obtained by the method are expressed by the relation between

dose of poison and the reciprocal of the survival period or speed of toxic action. Tervalent arsenic was found to be more toxic than quinquevalent arsenic. The minimum toxic dose of arsenic for the two species studied is about 0·02 mg. of arsenic per gram of insect. C. T. GIMINGHAM.

PATENTS.

Production of available phosphate. H. H. MEYERS, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,578,339, 30.3.26. Appl., 20.10.22).—A mixture of ground phosphate rock and an alkali salt is sintered, thereby rendering part of the phosphate available; the sintered mixture is broken into lumps and calcined at a suitable temperature, and for a suitable time to complete the conversion of the phosphate into available phosphate. C. T. GIMINGHAM.

Treatment of raw phosphates. M. PIERRON (F.P. 600,734, 11.10.24).—Raw phosphates rich in or mixed with calcium carbonate are roasted and slaked with water. The resulting product is easily distributed and assimilated. J. S. G. THOMAS.

Insecticide-fungicide. E. C. HOLTON, Assr. to SHERWIN-WILLIAMS Co. (U.S.P. 1,578,520–3, 30.3.26. Appl., [A, B] 8.3.24, [C, D] 12.1.25. [A] Renewed 5.2.26).—(A) An aqueous solution of barium polysulphide is treated with hydrofluoric acid, in presence of a substance to prevent crystallisation. The precipitate of amorphous barium fluoride and finely-divided sulphur is dried and powdered. (B) An alkaline-earth polysulphide is treated with a suitable acid substance to produce hydrogen sulphide, and a mixed precipitate of finely-divided sulphur and a substantially insoluble toxic substance. Additional sulphur is formed by acting on the hydrogen sulphide with sulphur dioxide. The product is dried and powdered (C, D) Calcium (or barium) polysulphide is treated with hydrofluosilicic acid to give a mixed precipitate of finely-divided sulphur and calcium (or barium) silicofluoride, which is dried and powdered. C. T. GIMINGHAM.

Means for combating plant pests. W. ILISCH (Swiss P. 109,831, 26.3.24. Conv., 4.4.23).—Juices or extracts of such plants as tomato, rhubarb, tobacco, poppy, digitalis, delphinium, etc. are supplied through the roots to plants to be protected. Suitable chemicals may be added to the liquids. C. T. GIMINGHAM.

Manufacture of meal for fertilising and other purposes, and for recovery of oil from fish or vegetable refuse and the like. S. HILLER (E.P. 215,765, 7.5.24).—See U.S.P. 1,489,940; B., 1924, 479

Arsenate of lead [insecticide]. H. B. GOODWIN, Assr. to LATIMER CHEMICAL Co. (Reissue 16,331, 20.4.26, of U.S.P. 1,322,008, 18.11.19. Appl., 8.11.21).—See B., 1920, 40 A.

Insecticide, sheep dip, and the like. P. J. FRYER, Assr. to McDOUGALL AND YALDING, LTD. (U.S.P. 1,583,681, 4.5.26. Appl., 29.11.24).—See E.P. 246,252; B., 1926, 293.

XVII.—SUGARS; STARCHES; GUMS.

Defecation of diffusion juice with dolomitic lime, and with the scums obtained by saturating the intermediate juice. W. KOHN (Z. Zuckerind. Czechoslov., 1926, **50**, 209—215, 217—221).—Dolomitic lime used for the defecation of diffusion juice gave satisfactory results provided the amount was less than 0·4% of the volume of the liquid, the mud obtained settling out in 30 min. to about 20% of the total height. When larger quantities were used, practically no subsidence of the precipitate occurred. Saturation scums from the carbonatation of the intermediate juice failed to produce a sufficient defecation of the diffusion juice. J. P. OGILVIE.

Increase of colour of first-product massecuites during boiling. F. HOFFMANN (Z. Ver. Deuts. Zucker-Ind., 1926, 91—98).—Measured by the extinction coefficient in a polarisation photometer with double blue filter, the total increase of colour from thick-juice (evaporator syrup) to first massecuite in a white beet sugar factory was found to average 47%, or from thick-juice to molasses, about 255%. J. P. OGILVIE.

Basic dyes as flocculating agents for approximate quantitative determination of colloids in sugar-house liquors. M. S. BADOLLET and H. S. PAINE (Int. Sugar J., 1926, **28**, 23—28, 97—103, 137—140).—On testing the colloids separated from sugar-house liquors in a cataphoresis apparatus, they were found to be electro-negative, and a method has been devised for their approximate determination on the principle of electrical neutralisation, using night-blue as flocculating agent, and maintaining the p_H of all the products examined constantly at 6·0. Tests on cane juice which had been submitted to different methods of clarification showed the iso-electric ratio (ratio of weight of dye in mg. to weight of solids in mg., the colloid content of which is electrically neutralised by the dye) thus obtained to vary in a generally parallel manner with the amount of colloids obtained directly by ultra-filtration. Caramel, and the water-reversible fraction of the colloids separated from various sugar-house liquors by ultra-filtration, showed only a slight tendency to flocculate when electrically neutralised under the authors' experimental conditions. J. P. OGILVIE.

Determination of reducing sugars volumetrically in the presence of excess sucrose. N. SCHOORL (Archief Sukerind. Nederl-Indië, 1925, **33**, 273—278; cf. B., 1925, 329, 518).—Using a 300-c.c. flask, a mixture of 25 c.c. of modified Luff's reagent (Benedict, B., 1909, 385) and the same volume of sugar solution is heated to boiling in 3 min., and maintained in ebullition for exactly 5 min., the flask having been connected with a reflux condenser. After cooling to room temperature, 3 g. of potassium iodide and immediately afterwards 25 c.c. of 25% sulphuric acid are added, and the liquid is titrated with 0·1N-thiosulphate. A control determination using the same amount of sucrose as was present in the 25 c.c. of assay solution was made under

exactly the same conditions, it being found that 10 g. gave a reduction corresponding to 0·33 c.c. of thiosulphate, that is as much as 1 mg. of invert sugar. In ordinary good refined sugar the content of reducing sugars by this method was found to be about 0·002%. This method is much more sensitive than any using Fehling's solution, and is well suited for accurate and rapid work in the refinery or sugar factory. J. P. OGILVIE.

Determination of reducing sugars volumetrically in presence of an excess of sucrose. N. SCHOORL (Int. Sugar J., 1926, **28**, 276—277).—See B., 1925, 518.

See also A., May, 502, **Mechanical liquefaction of starch** (PETIT and RICHARD). 535, **Iodometric determination of aldehyde sugars** (PAUCHARD).

Preservation of syrups. SABALITSCHKA and BÖHM.—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Manufacture of Yoghurt bacteria in tablet form. SCHEERMESSER (Pharm. Ztg., 1926, **71**, 499).—Yoghurt tablets, almost free from carbohydrates and albumin, may be thus prepared :—Triply centrifuged skim milk (fat content less than 0·1%) is treated with rennet (thereby destroying half the casein), the enzyme destroyed by heating, and the flocculent whey–casein mixture hydrolysed with trypsin, in alkaline medium, at 40—50°. The hydrolysis is interrupted before completion, and there should then be present only a small proportion of Siegfried's peptone, hardly any kyrine, and still less mono- and diamino-compounds. The trypsin is destroyed by neutralisation of the liquid with *N*-hydrochloric acid till Congo-paper of litmus-amphoteric reaction is turned slightly blue. The liquid is heated for some time at 80—90°, filtered, sterilised at 2-day intervals with steam, cooled to 40°, and inoculated with pure Yoghurt culture. Multiplication of the *Bacillus bulgaricus* is allowed to proceed in a thermostat, and may be hastened by the addition of certain salts. The addition of slight traces of certain oxides (acting catalytically) has a marked influence on the virulence of *B. bulgaricus*. The liquid is treated with 10% of a 10% calcium chloride solution, and then with an equivalent quantity of sodium pyrophosphate (both sterilised), when, on prolonged shaking, the precipitated calcium phosphate adsorbs the Yoghurt bacteria. The supernatant liquid is decanted and the residue centrifuged, powdered, and dried at 40°. The dry substance gives tablets containing only 1·3% of organic matter; 0·5 g. is sufficient to prepare 1 litre of Yoghurt milk. B. FULLMAN.

Volatile acidity of wines. C. LAGNEAU (Ann. Falsif., 1926, **19**, 152—159).—Duclaux's method of determining volatile acidity by titration of 50 c.c. of the distillate obtained from 20 c.c. of wine and 35 c.c. of water with 0·05*N*-sodium hydroxide solution was found satisfactory, but the use of the formula $0{\cdot}00245 \times n \times 50 \times 100/85$ is advised. Free sulphurous acid passes over completely into the distillate and must therefore be deducted from the total, whilst of the combined sulphurous acid a portion only, varying according to the conditions, distils over. D. G. HEWER.

Impurities in wines. J. VILAR (Anal. Asoc. Quím. Argentina, 1925, **13**, 337—347).—Analyses are given of a number of samples of wine collected in Argentina, some of which were sophisticated. Methyl alcohol is a normal impurity of wines which have been prepared by fermentation of grape juice in the presence of the skins and is formed at the end of the period of fermentation. G. W. ROBINSON.

Production of alcohol from rice straw. N. TAKETOMI (J. Soc. Chem. Ind. Japan, 1926, **29**, 43—47).—Rice straw was heated with 1% sulphuric acid at about 134° for 30 min. in an autoclave. The liquid obtained was neutralised with calcium carbonate, concentrated, and fermented with the distillery yeast, Race XII. The yield of sugar was about 15% of the dry matter used and of alcohol about 5·4%. K. KASHIMA.

Concentration of the alcohol content of alcohol-water vapour by separation of the aqueous distillate [phlegms]. J. DEHNICKE (Z. Spiritusind., 1926, **49**, 50—51, 71—72, 86, 101, 110).—If an alcohol–water vapour mixture is condensed by cooling, the phlegm has a lower alcohol content than the dephlegmated vapour. The difference between the amounts of alcohol contained in the phlegm and the dephlegmated distillate is greater, the smaller is the separated phlegm. The smaller the aqueous distillate, the lower is its alcohol content, and the greater the amount of aqueous distillate, the higher becomes the alcohol content of the dephlegmated vapour. The increase in the alcohol content of the dephlegmated vapour corresponding to increased phlegm formation tends to disappear with increasing alcohol content of the original vapour mixture. Slight increases have even been noticed with a 96% by volume vapour. Confirmation is given of the observation of Pampé that the use of the dephlegmator rarely brings about an increase of the end product with high-percentage alcohol vapours approximating to 96% by volume. Support is also given to the view of Hausbrand and Donitz that, by the condensation of vapour containing alcohol, there is obtained a vapour of higher and a phlegm of lower alcohol content than that of the original vapour. A flow of vapour through the dephlegmator so rapid that re-evaporation cannot be effected together with a slow current of cooling water produce less efficient concentration, since less phlegm is formed with accompanying poorer concentration of the vapour. The smaller production of phlegm is followed by a reduced re-evaporation. If the cooling surface is curtailed, the temperatures of the cooling water and cooling surface are low and the action of the dephlegmator is restricted. In this case, although the predominating factor is phlegm formation, its effect is insignificant. Modern

dephlegmators work as completely as possible on the principle of flow and counterflow of phlegm and vapour and are provided with a cooling surface which ensures phlegm formation without increasing the consumption of vapour over normal limits, and which are so regulated that the phlegm travels from cooler to warmer places, thus favouring re-evaporation. C. Ranken.

Influence of sugar on determination of ammonia in grape musts. J. Ventre and E. Bouffard (Ann. Falsif., 1926, **19**, 226—230).—See B., 1926, 417.

Souring of potatoes. Glaubitz.—See XIX.

Patents.

Preparation of fruit wines resembling grape wines in aroma and flavour. M. Hamburg (Austr. P. 97,133, 5.7.23).—By evaporation under reduced pressure, fruit and berry juices are freed almost completely from their volatile aromatic substances and are subsequently fermented with wine yeast. C. Ranken.

Acetone and similar compounds produced by fermentation. N. Moskovits (Austr. P. 100,444, 6.3.22. Conv., 11.3.21).—Reducing agents such as hyposulphites, thiosulphates, or sulphites are added to the mash together with the micro-organisms, *e.g.*, *Bacillus macerans*, which bring about the formation of acetone and similar substances. The favourable influence of the addition of sulphites is most marked. C. Ranken.

Production of fermentable worts. W. H. Campbell (U.S.P. 1,581,918, 20.4.26. Appl., 18.7.25).—See E.P. 237,321; B., 1925, 822.

XIX.—FOODS.

Adam's method of fat determination in milk. Chavastelon and Elouard (Ann. Falsif., 1926, **19**, 159—160).—The line of demarcation of the ethereal solution of the fat as obtained in Adam's method is more readily defined if four drops of an alcoholic solution (saturated when hot) of Sudan III. Red are added. The chromolipoid of the tomato or pumpkin may also be used. The use of the colouring matter resulted in an increase of 0·3—1·3 g. of fat per litre of milk over that obtained without its use. D. G. Hewer.

Determination of free acid and fat in technical casein. W. Höpfner and K. Jaudas (Chem.-Ztg., 1926, **50**, 325—326; cf. B., 1925, 420).—Polemical. The methods of determining free acid and fat in casein described by Ulex (B., 1925, 734) give too high results in each case, and Ulex's criticism of the authors' method is shown to be unsound. The proteins of casein absorb varying amounts of alkali according to the strength of solution used, so that erratic results are obtained by any method involving treatment of the whole with alkali. Treatment of the hydrochloric acid solution of casein with ether extracts not only fat, but also some lactic acid and certain degradation products of casein. A more satisfactory method for determining fat in technical casein consists in dissolving 2 g. in 10 c.c. of ammonia, shaking with 20 c.c. of 96% alcohol, then, after keeping for 20 min., with 10 c.c. of light petroleum and 20 c.c. of ether. An aliquot part of the ethereal layer is evaporated after 2 hrs., and the fat determined as usual. A. R. Powell.

Vitamin-A in oleo oil and oleo stearin. R. Hoagland and G. G. Snider (J. Agric. Res., 1926, **32**, 397—416).—The content of vitamin-A of a number of samples of oleo oil and oleo stearin was compared by feeding tests with young rats, the oil or stearin providing the only source of vitamin-A in an otherwise adequate ration. Various grades of oleo oil differed considerably in their content of this vitamin, the yellow oil being the richest. The samples of stearin also differed greatly, yellow stearin being richer than the other grades. Oleo oil is poorer in vitamin-A than butter, but richer than lard and vegetable fats and oils. C. T. Gimingham.

Drying of vegetables. A. W. Knapp (J.S.C.I., 1926, **45**, 123—128 t).—It is important to commence drying in an atmosphere of moderately high humidity to prevent case-hardening. The air should leave the plant at the highest temperature which is permissible without injury to the vegetable. The most efficient drying machine available during the war was the Tomlinson-Haas. It had the advantage that the air was heated between successive chambers, and also between successive batches of trays, so that it never became too humid. The properties of various vegetables and their preparation before drying are discussed. The yield is indicated by the specific gravity. Precautions must be taken to avoid the blackening of potatoes due to peroxydase. The loss on washing is from 0·3 to 0·5%, and on steaming 1%. White carrots are richer in sugar, but poorer in flavour than the red. The green colour of cabbage can be preserved by the use of ammonia. Onions should not be allowed to come in contact with copper. The moisture content of raw and dried vegetables, and of dried vegetables after exposure to the atmosphere is discussed in relation to yield and keeping properties. The yields of dry vegetables obtained from 100 pts. of the raw material were: potatoes 16 pts., carrots 10, onions 10, swedes 8, turnips 6, and cabbage 5. Dried vegetables have excellent keeping properties; they are best preserved in the dark and *in vacuo*.

Chemical, microscopical, and bacteriological study of infant foods. H. Kufferath (Chim. et Ind., 1926, **15**, 331—348).—An extensive analytical examination of typical infant foods is recorded, and is made the basis of a critical review of the feeding values of these preparations. The foods are classified according to the type of products used in their manufacture, and a number of fraudulent and deceptive practices adopted by manufacturers are discussed. A. G. Pollard.

Autolytic and bacterial transformation of fish muscle proteins. G. B. Reed (Pub. Health J.,

Canada, 1925, **16**, 568—577).—Preliminary experiments showed that there was a slight increase in non-coagulable and ammonia nitrogen in macerated haddock muscle kept under toluol at 25° for 24 hrs., and a conspicuous increase in 48 hrs. Haddock fillets were found to be heavily infected with bacteria similar to those of the gut and slime of the fish. When these bacteria were permitted to develop in fish muscle at 25° there were only slightly greater increases in non-coagulable and ammonia nitrogen during the first 24 hrs. than produced by autolysis alone, but from the twenty-fourth to the forty-eighth hour of incubation the bacteria produced very much greater protein transformation than was produced by autolysis alone. R. E. THOMPSON.

Legumin of sweet almonds. M. A. RAKUSIN and B. E. MASCHKILEISSON (Z. Unters. Lebensm., 1926, **51**, 45—47).—The comminuted, shell-free almonds, after removal of oil, sugar, and protein cleavage products by extraction with petroleum ether, alcohol, and water respectively, yielded, on extraction with sodium hydroxide solution, 4·8% of legumin having $[\alpha]_D$—42·90° (in pepsin-hydrochloric acid solution). The legumin gave colour reactions identical with those given by legumin from legumes (cf. preceding abstract). The optical rotations of the alkali salts of almond legumin are; *ammonium*, $[\alpha]_D$—64°; *lithium*, $[\alpha]_D$—39°; *sodium*, $[\alpha]_D$—42·53°; *potassium*, $[\alpha]_D$—34°. These resemble very closely those of the alkali salts of legumin from other sources (cf. B., 1923, 421). E. H. SHARPLES.

Souring of potatoes. M. GLAUBITZ (Z. Spiritusind., 1926, **49**, 53).—Potatoes intended for use as fodder are preserved by a souring process effected by means of a fermentation by lactic acid bacteria. The raw potatoes, after washing to remove undesired acetic and butyric acid bacteria, are sliced and packed as closely as possible into pits, preferably lined with cement. A liquid preparation of lactic acid bacteria is sprayed over the potatoes and the whole covered by boards or tiles to exclude air and rain which encourage mould formation. Frosted potatoes, owing to their sugar content, are specially suitable for preserving by this treatment. The potatoes may be steamed before addition of the bacterial preparation. The lactic acid bacteria preparation is made by inoculating, with a pure lactic acid bacteria culture, the pulp produced by boiling potatoes in a mixture of water and skimmed milk, and cooling to 30—35°. The author confirms the observations of Völtz on the relative values of raw, steamed, soured-raw, and soured-steamed potatoes as fodder for ruminants, and adds that soured-steamed potatoes can be safely given to horses and even to animals with young. C. RANKEN.

Identification of dried apple marc in foods. C. BRIOUX (Ann. Falsif., 1926, **19**, 142—144).—The epicarp or skin, endocarp or the hard parchment-like membrane bordering the spaces around the pips, and the testa or skin of the pips, have characteristic microscopical features (shown in plates). D. G. HEWER.

Black currant juice and reactions of orchil. M. FRANÇOIS and L. SEGUIN (Ann. Falsif., 1926, **19**, 230—235).—See B., 1926, 382.

Nutritive value of pasture. WOODMAN, BLUNT, and STEWART.—See XVI.

Yoghurt bacteria in tablet form. SCHEERMESSER.—See XVIII.

PATENTS.

Production of coffee free from caffeine. H. FAIRBROTHER. From F. KUNDIG, JUN., & Co., CHEM. FABR. (E.P. 247,039, 22.4.25).—See F.P. 597,392; B., 1926, 297.

Evaporating apparatus (E.P. 248,131).—See I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of santonin in *Artemisia* sp. and in resins. H. VOGTHERR (Arch. Pharm., 1926, **264**, 324—327).—The method of Fromme (Jahresber. Cæsar u. Loretz, 1912, 40) is suitably modified. For whole *Artemisia* plants 50 g. (finely powdered) are extracted with 500 c.c. of chloroform and the determination then completed as described by Fromme. For resinous substances further modification is necessary: the chloroform extract must be washed with dilute sodium hydroxide solution to remove resinous substances. With the quantities and strengths recommended the santonin removed at this stage is negligible. The finely powdered drug (10 g.) is stirred at 80—100° with water (50 c.c.) and hydrochloric acid (25%; 10 c.c. or more). The cake obtained is again ground and the whole mixture washed with water (40 c.c.) into a separating funnel, where it is extracted with chloroform (150 c.c. in three portions). The total chloroform extract is washed with sodium hydroxide solution (1·5%; 50 c.c.), then with water (50 c.c.), and after filtering over sodium sulphate, if necessary, is evaporated to small bulk (10 c.c.). The solution is covered with dilute barium hydroxide solution (5%; 100 c.c.) and the remaining chloroform boiled off. The liquor is filtered from resinous matter, acidified with hydrochloric acid (25%; 5 c.c.), and again extracted with chloroform (60 c.c., in 3 portions). The extract is filtered into a tared 100 c.c. flask, the chloroform is distilled off, the residue is dissolved in alcohol (7·5 g. exactly), and reprecipitated by adding hot water (42·5 g. exactly). After 24 hrs. the crystalline santonin is collected on a tared filter paper, washed twice with aqueous alcohol (3 pts. of alcohol to 17 of water; 10 c.c.), dried, and weighed. That remaining in the flask is also weighed. To the weight found 0·034 g. should be added. If in the final crystallisation there is a separation of resin the product must be washed with more aqueous alcohol and the weight suitably corrected. W. A. SILVESTER.

Commercial papain; its purification. R. FROSSARD (Bull. Soc. Chim. biol., 1926, **8**, 288—

293).—Comparison has been made of the yields, activity, and suitability for therapeutic use of the products of precipitation of the crude juice by alcohol, and by alcohol followed by dialysis and reprecipitation by alcohol. The single precipitation by alcohol yields a suitable active product.

R. K. Cannan.

Preservation of [pharmaceutical] syrups. T. Sabalitschka and E. Böhm (Pharm. Ztg., 1926, **71**, 496—498).—The action of methyl *p*-hydroxybenzoate ("Nipagin" or "Solbrol") and various other agents against the attack of *Aspergillus* and *Penicillium* and the like on various syrups has been tested. *Syrupus simplex* (60% of sugar), *S. mannæ* (55% of sugar), *S. althaeæ* (63% of sugar), and *S. rhei* (60% of sugar), all prepared according to D. A.-B. V., were treated with the various preservatives, which in the efficacy of their action against moulds could be arranged as follows:—Thymol=methyl *p*-hydroxybenzoate>benzoic acid=salicylic acid>Mikrobin (sodium *p*-chlorobenzoate)=sodium benzoate. To inhibit completely growth of mould required 0·05% of thymol, or 0·025—0·075% of methyl *p*-hydroxybenzoate, or 0·1—0·15% of benzoic acid. Methyl *p*-hydroxybenzoate is approximately equal to menthol in efficacy, whilst it has the advantage of no disturbing taste; it is superior to benzoic acid in being much less susceptible to the reaction of the medium, and, being practically neutral, it has no action on the substance preserved. It may also be used for the preservation of many other preparations (for internal and external use) against moulds and bacteria.

B. Fullman.

Preparation of acetylsalicylic acid, acetanilide, and acet-*p*-phenetidide. P. Manicke and P. Grigel (Arch. Pharm., 1926, **264**, 322—324).—Directions are given for quickly preparing the above compounds in the laboratory. Salicylic acid, aniline, or *p*-phenetidine (10 g.) is mixed with acetic anhydride (20, 15, or 10 g. respectively) and to the mixture, which becomes hot, a few drops of concentrated sulphuric acid are added. The interaction being complete, the pasty mass is stirred with water (200 c.c.). The product so obtained is either filtered off directly, or the suspension is boiled to cause the compound to dissolve; it crystallises out again on cooling. Almost the theoretical yield is obtained.

W. A. Silvester.

Mechanism of formation of *o*- and *m*-hydroxybenzaldehydes from the nitration product of benzaldehyde. H. H. Hodgson and H. G. Beard (J.S.C.I., 1926, **45**, 91—93 T).—Brady and Harris's statement (J.C.S., 1923, **123**, 484) that not much more than a 65% yield of pure *m*-nitrobenzaldehyde can be obtained from the mixed nitration product of benzaldehyde is confirmed. It follows that the optimum yield of *m*-hydroxybenzaldehyde, as well as the economy of any process adapted for its large-scale preparation, will depend on the separation of the reaction products given by the 20% of *o*-nitrobenzaldehyde present in the raw material. The best results were obtained by reducing the mixed *o*- and *m*-nitrobenzaldehydes to the amino-compounds, and converting the latter into the hydroxy-compounds by diazotisation and subsequent decomposition in a current of steam. The *o*-hydroxybenzaldehyde is volatile in steam and hence is continuously separated from the *m*-hydroxybenzaldehyde. In a mixture of *o*- and *m*-nitrobenzaldehydes sodium hyposulphite reduces the latter to *m*-aminobenzaldehyde and the former to *o*-aminobenzaldehyde, possibly via anthranil. Addition of hyposulphide in more than one batch considerably decreases the amount of anthranil ultimately formed. The amines, however, must be immediately diazotised after formation to diminish the loss entailed by their rapid decomposition, which appears to commence at formation, and the sulphuric acid present must be ample to prevent oxime or hydrazine and triazole formation.

Formaldehyde-sodium bisulphite and formaldehyde-sulphurous acid. Determination and properties. W. Ledbury and R. Taylor (J.S.C.I., 1926, **45**, 85—89 T).—The hypoiodite method of Romijn for the determination of formaldehyde (Analyst, 1897, **22**, 221) gives satisfactory results when applied to the determination of formaldehyde-sodium bisulphite, and since formaldehyde-sodium bisulphite is not acted upon by iodine in acid or neutral solution any uncombined sodium bisulphite present can be determined by direct titration with iodine. Complete combination between formaldehyde and sodium bisulphite in solution does not occur at ordinary temperatures, even on prolonged keeping, but is attained in a few minutes by warming at 70°. Formaldehyde-sulphurous acid can be determined in solution in the presence of uncombined formaldehyde by a direct determination of the acidity, and from the total iodine value of the solution it is possible to determine uncombined formaldehyde. When formaldehyde-sulphurous acid occurs in solution in the presence of sulphurous acid, its concentration can be derived from the acidity of the solution if allowance is made for sulphurous acid, which can be determined iodometrically. Distillations of solutions containing formaldehyde-sulphurous acid with either formaldehyde or sulphurous acid in excess, give rise to distillates and residues containing formaldehyde-sulphurous acid and uncombined formaldehyde, but no sulphurous acid.

Essential oil of manuka (*Leptospermum scoparium*). W. F. Short (J.S.C.I., 1926, **45**, 96—98 T; cf. Gardner, J.S.C.I., 1924, **43**, 34 T; 1925, **44**, 528 T; Inder, Thesis New Zealand Univ., Nov., 1923).—1064 lb. of fresh leaves from Birkenhead, Auckland, N.Z., gave on steam-distillation a 0·37% yield of a greenish-brown oil: d_4^{25} 0·9179, n_D^{25} 1·4952, $[\alpha]_D^{25}$—16·8°; soluble in 10 vols. of 90% alcohol and in 40 vols. of 80% alcohol. The percentage composition of the oil is as follows: α-pinene (12%), eudesmene and a second sesquiterpene (60%), citronellol and geraniol both free and as their *iso*valeric, cinnamic, and acetic esters (6%), citronellal and citral (1%), cineole (1%), leptospermol 8%, azulene (traces), *iso*valeric, cinnamic, and acetic acids, free and combined (4%). Leptospermol

does not occur in the sodium bicarbonate extract, and Gardner's statement (*loc. cit.*) that it is an acid and not a phenol is probably incorrect. It is probable that the sesquiterpene "manukene" of Gardner, giving a liquid monohydrochloride, is impure eudesmene, since if the crude eudesmene hydrochloride is distilled at pressures above 4 mm., the distillate has approximately the composition $C_{15}H_{25}Cl$.

See also A., May, 468, **Influence of sodium hydroxide on adsorption of arsenious acid by "saccharated" iron** (HERBOTH). 490, **Determination of arsenic in organic compounds** (TER MEULEN). 492, **Determination of mercury in organic and inorganic compounds** (TER MEULEN). 497, **Basis for physiological activity of certain -onium compounds. Sulphur analogue of choline** (RENSHAW, BACON, and ROBLYER). 499, **Equilibrium between lactic acid and anhydride in commercial lactic acid** (EDER and KUTTER). 500, **Selective reduction of citral** (ADAMS and GARVEY). 516, **Synthesis of hydroxydivarinol** (MAUTHNER). 517, **α-Naphthylcarbimide as a reagent of alcohols** (BICKEL and FRENCH); **Preparation of ethers from aromatic alcohols** (SENDERENS). 518, **Hydrolysis of saccharin** (TÄUFEL and NATON). 520, **Catalytic hydrogenation of the carbonyl group in aromatic compounds** (KUBOTA and HAYASHI). 522, **α- and β-Amyrin** (DISCHENDORFER and RENDI); **Saponins and related substances. Æscigenin** (VAN DER HAAR).

PATENTS.

Removing nicotine from tobacco. T. SCHLOESING (E.P. 238,517 and 242,225, 29.5.25, Conv., [A] 18.8.24, and [B] 31.10.24. Addns. to 234,845).—(A) In the process described in the chief patent (B., 1926, 465), a substantially constant ammoniacal pressure was permanently maintained. It is now shown that if the ammonia pressure be maintained at a pressure of a certain order, varying, *e.g.*, between 25 g. or less, and 160 g. or more of gaseous ammonia per kg. of steam, then the elimination of nicotine proceeds rapidly and depends only on the quantity of steam passing. The proportion of ammonia need not be carefully regulated.

(B) The kerosene or other solvent used to recover the nicotine extracted as described in the chief patent dissolves some ammonia, which hinders the recovery of nicotine. The ammonia may be removed by passing the solvent through water (if necessary, saturated with a salt such as sodium chloride, which checks dissolution of nicotine); or by bubbling a gas such as air through the solvent, or by passing a current of the gas over the solvent, which is finely divided by causing it to fall in thin sheets or drops, or to flow over solid materials such as pebbles.

B. FULLMAN.

Preparation of salts of *N*-halogen-substituted arylsulphonamides. CHEM. FABR. VON HEYDEN A.-G. (G.P. 422,076, 24.5.24).—Solutions of alkali or alkaline-earth salts of *N*-halogen-substituted arylsulphonamides (cf. G.P. 390,658; B., 1924, 811) are treated with soluble magnesium salts. For example on stirring an aqueous solution of calcium *p*-toluene sulphochloramide with an aqueous solution of magnesium chloride, magnesium *p*-toluenesulphochloramide separates in crystals containing 23·6% of active chlorine It is stable and is sufficiently soluble in water to be used for preparing disinfecting solutions. Similarly the magnesium salt of β-naphthalenesulphochloramide is obtained from sodium β-naphthalenesulphochloramide and magnesium chloride, and the magnesium salt of tetrahydronaphthalenesulphochoramide from the corresponding sodium salt (cf. G.P. 377,367; B., 1923, 1149 A) and magnesium sulphate. The products are used as disinfectants and for other purposes. A. COULTHARD.

Production of heterocyclic compounds containing arsenic or antimony. A. BINZ and C. RÄTH (E.P. 250,287, 30.10.24).—Heterocyclic compounds containing arsenic or antimony are prepared by the interaction of diazotised amino-derivatives of pyridine, quinoline, or *iso*quinoline with arsenites or antimonites or the corresponding free acids. The parent materials may contain substituents such as the hydroxy-, amino-, carboxy-, and phenyl radicals or halogens. The salts of these arsenic or antimony compounds are markedly non-poisonous. It is best to use 2—3 mols. of the arsenite or antimonite to each mol. of the original heterocyclic compound. Reduction of the heterocyclic arsinic or antimonic compounds with, for example, hyposulphites, hypophosphorous acid, or bisulphites converts them into arsenoxides of the type, RAs:O, and further reduction into arseno-compounds of the type, RAs:AsR. These with oxidising agents such as hydrogen peroxide, yield the original arsinic or antimonic acids in a purer form. For example, a diazotised solution of α-hydroxy-β′-aminopyridine is added, drop by drop, to a solution of sodium arsenite. The mixture is made feebly alkaline and kept for some time to complete the reaction. The α-hydroxy-β′-pyridinearsinic acid obtained from the solution as colourless crystals decomposes at 215°. When heated moderately with hypophosphorous acid, preferably out of contact with air, the arsinic acid gives αα′ββ′-dihydroxydiarsenopyridine, a pale yellow powder. This substance on treatment with cold 3% hydrogen peroxide gives the original acid but free from impurities. A. COULTHARD.

Synthesising higher molecular organic compounds containing oxygen. BADISCHE ANILIN- & SODA-FABR., Assees. of G. AND R. WIETZEL (U.S.P. 1,562,480, 24.11.25. Appl., 25.2.25).—By passing a vaporised aliphatic alcohol mixed with carbon monoxide at elevated temperatures and pressures over a catalyst containing hydrogenating constituents such as copper, silver, gold, tin, lead, or the like, and hydrating constituents such as an oxide of titanium, zirconium, thorium, vanadium, manganese, etc., products containing mixtures of higher alcohols, aldehydes, and esters are obtained. It is of advantage to add an alkali or alkaline-earth metal compound to the catalyst, and if ammonia be added to the carbon monoxide, compounds containing nitrogen are also produced.

The molecular weights and boiling points of the products vary with the time of contact, with the catalyst, and with the temperature. For example, a mixture of methyl alcohol and carbon monoxide is passed at 420° and 200 atm. over a catalyst containing an alkali, chromium, and molybdenum, when a mixture of higher alcohols, aldehydes, and esters derived from valeric and butyric acids and the like is obtained. Ethyl alcohol passed with carbon monoxide free from iron carbonyl at 400° and 200 atm. over a catalyst consisting of zinc oxide, chromium and manganese oxides contained in a tube lined with copper yields products which boil for the most part between 100° and 250°. T. S. WHEELER.

Manufacture of acetic anhydride and acetaldehyde from ethylidene diacetate. M. J. MARSHALL and G. S. SHAW, Assrs. to CANADIAN ELECTRO PRODUCTS CO., LTD. (U.S.P. 1,578,454, 30.3.26. Appl., 26.1.20).—If 1·5% of phosphoric acid be added slowly to boiling ethylidene diacetate decomposition to acetic anhydride and acetaldehyde according to the reaction $CHMe(OAc)_2=Ac_2O+Me\cdot CHO$ proceeds smoothly with but little formation of acetic acid. 1000 g. of ethylidene diacetate treated in this manner yield 238 g. of acetaldehyde, 555 g. of acetic anhydride, 71 g. of acetic acid, and 126 g. of unchanged ethylidene diacetate. T. S. WHEELER.

Producing dialkyl selenides and tellurides. C. A. HOCHWALT, Assr. to GEN. MOTORS CORP. (U.S.P. 1,578,731, 30.3.26. Appl., 3.10.23).—An alkali metal alloy of selenium or tellurium is treated with an alkyl sulphate to obtain the corresponding dialkyl selenide or telluride. For example, 960 pts. of the alloy Na_2Se are finely divided and treated at 100° with 1000 c.c. of ethyl sulphate, water is added, and the diethyl selenide formed is recovered in satisfactory yield by distillation in steam.
T. S. WHEELER.

Preparation of esters of formic acid. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of O. ERNST and K. SPONSEL (G.P. 422,500, 9.3.23).—Carbon monoxide, in the presence of water, is allowed to act on alcohols at raised temperatures with or without increased pressure and catalysts. Methyl formate is formed below 200° under ordinary pressure on leading carbon monoxide, methyl alcohol vapour, and steam over a variety of contact substance including such as favour the taking up and splitting off of water, but the reaction is greatly accelerated by increased pressure. Excess of alcohol improves the yields. For example, a mixture of carbon monoxide and methyl alcohol vapour in the proportion 1 : 2 with 0·1 vol. of steam when once led over titanium or thallium oxide, under ordinary presssure, at 175°, gives 0·1% of methyl formate. Using zinc and manganese as contact substances with highly activated carbon as carrier, the yield is 1·0% at 260°. With activated carbon at 200° and 200 atm. pressure, the yield of methyl formate rises to 12%. Similar results are obtained with other alcohols. A. COULTHARD.

Manufacture of carbamide. BADISCHE ANILIN- & SODA-FABR., Assees. of W. MEISER (G.P. 422,525, 26.4.22).—Ammonia is caused to react with carbon monoxide under pressure in apparatus lined with lead. The outside wall of the reaction vessel is rigid and has one, or preferably several, small openings, through which any air between the walls, which would otherwise cause folds in the lead on warming, can escape. Leakages through the lead itself can be detected by the hissing and smell of the gases. With these arrangements it is not possible for the melt, should the lead lining be destroyed, to corrode the outer wall and cause an explosion without warning. For example, in iron tube of 48 mm. inner diameter, provided with several holes less than 1 mm. diameter, has a lead tube of 32 mm. inner diameter introduced and pressed on to it. The simplest way of obtaining the openings is to screw rather loosely-fitting plugs into larger holes. A. COULTHARD.

Preparation of a *Bz*-tetrahydrohydroxyquinoline. J. D. RIEDEL A.-G. (G.P. 423,026, 10.8.23). — 6-Hydroxy-2 : 4-dimethylquinoline is treated with molecular hydrogen in the presence of a catalyst containing nickel. For example, hydrogen under pressure is allowed to act on 6-hydroxy-2 : 4-dimethylquinoline dissolved in decahydronaphthalene at 180—190° in the presence of nickel. The mixture is diluted with alcohol, filtered, and the solvents distilled off, leaving a crystalline substance insoluble in alkali. 5 : 6 : 7 : 8-*Tetrahydro-6-hydroxy-*2 : 4-dimethylquinoline, $C_{11}H_{15}ON$, has m.p. 162°, b.p. 181—183°/15 mm., gives no reaction with nitrous acid; the *hydrochloride* has m.p. 134°, *picrate*, m.p. 150°, *methiodide*, $C_{12}H_{18}ONI$, m.p. 140°. The O-*benzoyl* compound, m.p. 199—200°, made by warming the base in chloroform solution with benzoyl chloride, is an effective anæsthetic. A. COULTHARD.

Preparation of dicyclic bases. J. D. RIEDEL A.-G. (G.P. 423,027, 14.3.24).—Tetrahydrogenated dicyclic bases having more than three carbon atoms in the hydrogenated heterocyclic nucleus, are prepared by condensing β-phenylethylamine or γ-phenylpropylamine, or their derivatives, with α-halogencarboxylic acids or their esters, and with acid halides, treating the products with condensing agents, such as aluminium chloride or zinc chloride, to cause ring formation with splitting off of carbon monoxide and hydrogen chloride, yielding *N*-acyl derivatives of dicyclic bases, from which the acyl residue is split off by hydrolysis. For example, ethyl phenylethylglycine, b.p. 157—159°/12 mm., obtained by the action of ethyl bromoacetate on β-phenylethylamine, on evaporation with hydrochloric acid yields phenylethylglycine, $CH_2Ph\cdot CH_2\cdot NH\cdot CH_2\cdot CO_2H$, m.p. 244°; this compound on treatment with benzenesulphochloride yields *N*-benzenesulphophenylethylglycine, m.p. 122°, which on heating with phosphorus pentachloride and aluminium chloride in the presence of nitrobenzene as solvent, yields *N*-benzenesulphotetrahydro*iso*quinoline, m.p. 154°, and this on hydrolysis yields tetrahydro*iso*quinoline. 6-Methyltetrahydro*iso*quinoline, m.p. 255—256°, is obtained by treating the *N*-benzenesulpho-derivative of *m*-tolylethylglycine hydrochloride, m.p. 212—214° (obtained from *m*-methylphenylethylamine and

chloroacetic acid) with phosphorus pentachloride and aluminium chloride, and hydrolysis of the product, and homotetrahydro*iso*quinoline is obtained from γ-phenylpropylglycine, m.p. 201°, by a similar process. L. A. COLES.

Manufacture of poly-iodine substituted isatins. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 244,443, 23.11.25. Conv., 10.12.24).—A mixture of 5 : 7-di-iodoisatin, decomp. 234°, and 4 : 5 : 6 : 7-*tetraiodoisatin*, decomp. 190°, is formed when 2 mols. of iodine monochloride react with 1 mol. of isatin in concentrated mineral acid (*e.g.*, hydrochloric acid) solution. After 1 hr. the liquid is poured into ice water. After 1 day the precipitate is filtered off, steam distilled, the residue dissolved in excess of 0·5% sodium hydroxide, and the mixed iodo-compounds are precipitated by addition of sulphurous acid. Separation is effected by crystallisation from alcohol. The substances are of value for the preparation of pharmaceutical compounds. B. FULLMAN.

Manufacture of a new iodine substituted oxindole. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 244,444, 23.11.25. Conv., 10.12.24). —*Iodo-oxindole*, decomp. 168°, containing iodine in the nucleus, is thus prepared : a solution of 4·5 g. of oxindole in 100 c.c. of concentrated hydrochloric acid is added to 10 g. of iodine monochloride in 50 c.c. of concentrated hydrochloric acid. After 2 hrs. the mixture is poured into 3 litres of ice water. After 1 day the precipitate is filtered off, steam distilled, the residue dissolved in very dilute sodium hydroxide, and the iodo-oxindole precipitated by addition of sulphurous acid, and crystallised from alcohol. It is used in the preparation of pharmaceutical substances. B. FULLMAN.

Manufacture of aromatic stibinic acids. CHEM. FABR. VON HEYDEN A.-G. (E.P. 244,746, 4.12.25. Conv., 18.12.24).—The preparation of aromatic stibinic acids (*e.g.*, phenyl-, *p*-chlorophenyl-, and *m*-chloro-*p*-acetamidophenyl-stibinic acids) by the action of antimony oxide on the appropriate diazotised amino-compound is greatly facilitated by the presence of glycerol or other polyhydric alcohols, the formation of by-products being largely inhibited, while the acid formed is easily isolated. B. FULLMAN.

Manufacturing barbituric acid derivatives. L. THORP, Assr. to LAMBERT THORP CO. (U.S.P. 1,576,014, 9.3.26. Appl., 20.3.25).—5 : 5-Dialkylbarbituric acids are prepared by condensing dialkylmalonic esters with carbamide in an absolute alcoholic solution of sodium ethoxide and distilling off the alcohol without preliminary refluxing. Satisfactory yields are thus obtained without the use of elevated temperatures and pressures such as have hitherto been considered necessary. For example, ethyl ethyl*iso*propylmalonate is mixed with carbamide in an ethyl alcoholic solution of sodium ethoxide, the alcohol distilled off at 107°, and the sodium salt of 5 : 5-ethyl*iso*propylbarbituric acid which remains is treated with hydrochloric acid to yield the free 5 : 5-ethyl*iso*propylbarbituric acid, m.p. 200—201° corr., of therapeutic value as a sedative. T. S. WHEELER.

Preparation of aminoketones. C. MANNICH (G.P. 422,098, 30.11.19).—Aminoketones are prepared by the action of mono- or di-alkylamines and formaldehyde, or condensation products of alkylamines and formaldehyde, upon *cyclo*ketones. For example, an aminoketone, $MeC_6H_8O.CH_2.NMe_2$, a colourless oil, b.p. 104°/20 mm. (cf. Mannich and Braun, A., 1920, i., 850), is obtained by heating 4-methyl*cyclo*hexanone with dimethylamine hydrochloride and 30% formaldehyde for several hours. The ketones and aromatic acid esters of their reduction products are of therapeutic value as anæsthetics. L. A. COLES.

Preparation of organic derivatives of tervalent vanadium. FARBENFABR. VORM. F. BAYER & CO. (Austr. P. 101,685, 28.7.24).—The compounds, which are of therapeutic value, are obtained by the action of vanadium trioxide or its salts on salts of polybasic organic acids, of hydroxy-acids, or of hydroxy-thioacids, or, alternatively, by treating the free acids with vanadium trioxide or its salts, and subsequently neutralising the products with alkali or alkaline-earth hydroxides or carbonates. Examples are given of the preparation of complex sodium–vanadium compounds of nitrolotriacetic acid (cf. U.S.P. 1,517,003 ; B., 1925, 115), of tartaric acid, of thioglycollic acid, of salicylic acid, and of mandelic acid. L. A. COLES.

Separation of hexamethylenetetramine from ammonium chloride. H. WADE. From S. KARPEN & BROS. (E.P. 246,415, 23.10.25).—See U.S.P. 1,566,821 ; B., 1926, 217.

Production and separation of hexamethylenetetramine and ammonium chloride. H. WADE. From S. KARPEN & BROS. (E.P. 246,416, 23.10.25). —See U.S.P. 1,566,822 ; B., 1926, 217.

Arsenical albumin compound. O. BALLY, Assr. to HACO-GES. A.-G. BERN (U.S.P. 1,581,826, 20.4.26. Appl., 23.11.23).—See E.P. 208,143 ; B., 1925, 189.

Injectable solutions of drugs insoluble in water. SOC. OF CHEM. IND. IN BASLE, Assees. of E. S. FAUST (U.S.P. 1,582,456, 27.4.26. Appl., 15.6.23).—See E.P. 211,771 ; B., 1924, 400.

Manufacture of glutamic acid and salts thereof. K. IKEDA (U.S.P. 1,582,472, 27.4.26. Appl., 5.1.25).—See E.P. 248,453 ; B., 1926, 420.

Production of formamide. BADISCHE ANILIN- & SODA-FABR., Assees. of R. FICK (U.S.P. 1,582,675, 27.4.26. Appl., 3.4.25).—See E.P. 237,528 ; B., 1925, 827.

Preparation of hydroxy aliphatic arsonic acids. C. OECHSLIN, Assr. to ETABL. POULENC FRÈRES (U.S.P. 1,582,775, 27.4.26. Appl., 25.10.23). —See E.P. 206,152 ; B., 1925, 83.

Preparation of substituted aliphatic arsonic acids. C. Oechslin, Assr. to Etabl. Poulenc Frères (U.S.P. 1,582,776, 27.4.26. Appl., 25.10.23). —See E.P. 206,143 ; B., 1924, 768.

Pharmaceutical product. [Compound of phenylethylbarbituric acid and 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone.] Chem. Fabrik auf Actien (vorm. E. Schering), Assees. of A. Thiele (U.S.P. 1,582,802, 27.4.26. Appl., 18.5.23).—See E.P. 198,379 ; B., 1924, 114.

Silver-protein preparation. L. Taub, H. Janssen, and G. Wesenberg, Assrs. to Winthrop Chemical Co., Inc. (U.S.P. 1,582,940, 4.5.26. Appl., 11.7.22).—See G.P. 392,038 ; B., 1924, 578.

Fluid for industrial purposes (U.S.P. 1,575,967). —See I.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Conditions governing the behaviour of silver bromide grain during development. II. L. F. Davidson (Phot. J., 1926, 66, 230—242 ; cf. B., 1925, 151).—Sensitometric data, showing the variations due to the use of different developing formulæ, are given for plates of fast, "special rapid," and ordinary contrasty types, using as developers quinol–caustic soda, pyro–metol, pyro–soda, borax metol–quinol, and *p*-aminophenol. The course of the γ-time of development curves varies markedly for a given plate with the developers used, and the γ_{∞} values vary greatly. Using single-layer plates, the form of the developed grain was studied. Pyro–soda, pyro–metol, and quinol produce the same form of developed grain, with sharply-defined edges, and the original shape is retained to a certain extent. Borax M.Q. gives irregular, feathery grains with thin, diffuse edges, and some grains only partially reduced. *p*-Aminophenol gives a more irregular, spongy grain, all sign of original shape being lost, and some grains are only partially developed ; large numbers of single grains remain undeveloped, Borax M.Q. and *p*-aminophenol both give low γ values. Previous results concerning the induction period, and that grains of fast plates show a bigger percentage size increase on development than those of slower plates, are confirmed. The measurements were repeated, plotting the size–frequency curves of the same grains before and after development. Certain size–frequency curves showed a secondary hump after the maximum, on the side of increasing grain size. The curves for the developed grains show the same general characteristics as those for the undeveloped, but there is an increase in range, and a shift in position varying in amount with the developer and plate used, and a new hump at the small grain size end of certain curves, in addition to the two humps in the original curves. The causes of density variation with different developers are discussed.

W. Clark.

Photochemical studies. VII. Physical development of the latent image. A. Reychler (Bull. Soc. chim. Belg., 1926, 35, 74—80 ; cf. B., 1925, 265).—Physical development of a bleached and fixed negative and of the true latent image is considered. Ten plates, 9 by 12 cm., exposed and fixed preparatory to physical development by Lumière's method, were stripped and ashed, and an appreciable amount of silver, representing the latent image, was determined. The average number of silver atoms of the latent image per unit area of plate can be determined in this way. With a plate exposed, bromided (with chromic acid–bromide), and washed, and then fixed, a good image can be obtained by physical development with the mercury formula. If the plate is bromided after fixation, only very poor images can be physically developed. These facts indicate that in any grain there are numerous latent image nuclei distributed throughout the body of the grain, but only those on the surface are attacked by the bromiding mixture before fixing. Light acts not only on the surface but in the interior of the grains, and on those molecules of silver bromide of which decomposition is facilitated by a neighbouring gelatin molecule or group of molecules which act as bromine acceptors. The emulsion is a diphase system in which the gelatin contains colloidally dispersed silver bromide, and the grains have occluded gelatin ; in both cases the substances are intimately mixed and may be chemically united

W. Clark.

Photochemical studies, VIII. Chemical development of the latent image. A. Reychler (Bull. Soc. chim. Belg., 1926, 35, 80—82 ; cf. preceding abstract).—Results with chromic acid solutions indicate that in chemical development only the latent image nuclei on the grain surfaces are effective. Treatment of a plate in 2% chromic acid is stated not to alter its photographic properties to any great extent. Albert's process for direct positives is discussed. The reason that a latent image nucleus, destroyed by chromic acid, is not formed again at the same point on subsequent exposure, is ascribed to the fact that the silver bromide molecules which give rise to the first nucleus are in the neighbourhood of some part of a gelatin molecule (*e.g.*, $CO{\cdot}NH_2$ or $CH{\cdot}NH_2$) which readily takes up bromine. When these groups are saturated with bromine, they cannot function in a subsequent exposure. Physical development can be prevented by prolonged treatment of the exposed grains with chromic acid, or by a brief action of chromic acid after fixing out the silver bromide.

W. Clark.

Photographic spectrophotometry. Kellner. —See A., May, 483.

Patents.

Kinematograph multi-colour films. J. E. Thornton (E.P. 244,571, 18.5.23).—Raw film stock of the screen-filter type, upon which the component images of a built-up positive film are printed, or upon which the negatives used in printing these images are produced, is made in the form of strips, each of which comprises a celluloid base having a two-colour filter layer formed thereon in

dot, line, or mosaic pattern, and a panchromatic gelatino-silver emulsion coated upon the filter layer, the colours corresponding each to one component of the four- or three-colour picture. The negative film stock is of standard thickness (0·006 in.), and the positive film stock is of half standard thickness. The screen layers are produced by coating the base with an adhesive substratum on which grains of starch, gelatin, resin, etc., dyed in one colour are scattered and rolled in, and dyeing the inter-spaces with the second colour; by coating the base with a layer of gelatin or albumin dyed in one colour and sensitised with dichromate, printing under a dot, line, etc. screen, washing to remove dye from the unprinted parts, and dyeing the interspaces with the other colour; or by a similar process but using a film sensitised with silver salts.

Kinematograph and other colour films. J. E. THORNTON (E.P. 246,257, 17.11.24. Addn. to 224,569–70, 230,965, 233,990, and 231,030; cf. E.P. 213,866 and 224,571, B., 1924, 538, and preceding abstract).—In a multicolour kinematograph positive film of the type described in the previous patents, the two thin films are cemented with the image-face of one to the bare celluloid or unprinted bare gelatin back of the other. This enables all the positive images to be printed from negatives in which all the component images face the same way, none being reversed. W. CLARK.

Multicolour kinematograph or other films. J. E. THORNTON (E.P. 246,266, 25.11.24. Addn. to 224,569).—Double-width film of half standard thickness carrying a sensitised colloid is printed with two side-by-side rows of colour component positive images from a double-width negative, finished, and dried. The printed surface is then re-coated with a sensitiser and two further rows of images are printed, superimposed on the first. The film is then severed longitudinally and the two strips are superimposed in register and cemented. The first pair of prints may be made by a silver sensitising process and the superimposed second pair by a dichromate sensitising process, or by using salts of iron, uranium, etc. Alternatively, the first pair of prints may be made by a dichromate method and the superimposed pair by a sensitising process using other metal salts, such as those of silver, iron, uranium, etc.; or both first and second pairs of prints may be made by the same process, and in relief and/or non-relief, or the first pair of prints may be made by a sensitised colloid process, and superimposed by a second pair produced photomechanically without light action on an insensitive surface. W. CLARK.

Kinematograph and other positive films. J. E. THORNTON (E.P. 246,282, 19.12.24).—For preparing a scratchless film of the enclosed-image type, the two printed colloid faces of the separate thin films are treated (in some cases after partially softening the colloid faces) with exceedingly thin layers of cellulose cement which is allowed partially to penetrate. The two moistened surfaces are then united by pressure. The printed faces of both films may be roughened or toothed, then treated with dilute cellulose cement, and finally pressed together. The colloid faces may alternatively be both roughened and softened before cement is applied. The cellulosic cement consists of a cellulose compound such as celluloid, cellulose acetate, etc., a suitable solvent, *e.g.*, amyl acetate, acetone, etc., and a solvent or partial solvent of the gelatin or other printed colloid used, *e.g.*, glacial acetic acid. To this cement may be added for greater flexibility a colloid such as gelatin, glue, or fish-glue in a solvent such as glacial acetic acid, and a substance such as glycerin, syrup, or sugar, for increasing the flexibility. W. CLARK.

Kinematograph and other colour positives. J. E. THORNTON (E.P. 246,283, 19.12.24).—The material for preparing multicolour transparent positives consists of two different kinds of image-layers for producing two different kinds of images. One layer is sensitised with a panchromatic gelatino-silver bromide emulsion for giving temporary negative black images; the other layer is unsensitised and consists of interspersed fine lines, dots, etc., of coloured colloid for giving multicoloured permanent positive images. The two layers are on opposite sides of the same double-width support, or else superimposed on the same side, the silver layer being over the colloid layer. The silver layer covers the whole double-width film, while the colloid layer forms two parallel stripes each containing two colours, those of one being different from the colours of the other stripe. After treatment the printed material is divided and the two parts are superimposed and cemented face-to-face, back-to-back, or back-to-face. In printing, the silver emulsion layer is exposed through a pair of positives of the double-filter-screen side-by-side type, each filter consisting of two colours; the images are developed, fixed, washed, and dried; the multicoloured colloid layer is sensitised with dichromate, dried, exposed to light through the previously-formed silver images, and developed in hot water, and the black silver image is then dissolved away. W. CLARK.

[Non-inflammable] cellulose acetate composition [photographic film]. L. J. MALONE and S. J. CARROLL, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,575,778, 9.3.26. Appl., 2.8.20).—Flexible transparent films, free from inflammable nitrocellulose, are obtainable from compositions prepared by mixing a solution of cellulose acetate in acetone with castor oil which has been previously incorporated with a substance such as acetylene tetrachloride, methyl alcohol, methyl salicylate, or tricresyl phosphate, having the property of inducing transparency in the film. T. S. WHEELER.

Manufacture of photographic films, papers and the like. KODAK, LTD., W. G. BENT, and R. E. CROWTHER (E.P. 250,770, 5.5.25).—A medium, *e.g.*, an aqueous solution containing 0·25% of citric acid and 0·1% of Phenosafranine, which reduces the photo sensitiveness of the emulsion, and/or

prevents the subsequent development of any latent image immediately below or above the medium, is applied to the surface of the support of a photographic film, etc., or to the sensitised coating thereof. The method is applicable more especially to films, etc., upon which rulings intended to indicate the scale of the photograph are to be marked. J. S. G. THOMAS.

Light-sensitive medium and process of producing it. M. C. BEEBE, Assr. to WADSWORTH WATCH CASE Co. (U.S.P. 1,574 356, 23.2.26. Appl., 8.3.22).—The light-sensitive varnish-like medium prepared as described in E.P. 203,285 (B., 1924, 493) can be used as a resist for etching purposes and renders practicable the direct reproduction of a photographic image on metal. T. S. WHEELER.

Photographic print-out paper. J. A. JOHNSON (U.S.P. 1,582,050, 27.4.26. Appl., 25.4.21; cf. B., 1923, 1103A).—An emulsion of a silver compound other than silver iodide is treated with an iodide solution to form a silver iodide emulsion, and then with a sensitiser to make the silver iodide sensitive to light. W. CLARK.

Treatment of waste cinematographic films. D. B. MACDONALD (U.S.P. 1,582,847, 27.4.26. Appl., 6.5.22).—See E.P. 191,091; B., 1923, 203 A.

XXII.—EXPLOSIVES; MATCHES.

See A., May, 480, **Comparison between explosion temperatures calculated from specific heats and from explosion pressures** (MURAOUR). 503, **Nitration of cellulose** (CARRIÈRE).

PATENT.

Drying smokeless powders. J. C. VICKERY and M. H. DAVIS (U.S.P. 1,579,262, 6.4.26. Appl., 29.2.24).—A gaseous medium, previously partly saturated with the solvent by means of a spray, is heated, circulated over the powder, restored to its condition of partial saturation, and recirculated through the system. This cycle of operations is repeated until the powder has reached the desired degree of dryness. D. J. NORMAN.

XXIII.—SANITATION; WATER PURIFICATION.

Detection and determination of free chlorine in drinking water. I. M. KOLTHOFF (Chem. Weekblad, 1926, **23**, 203—204).—To 100 c.c. of the water acidified with acetic acid are added sodium acetate solution and a few drops of a 0·1% solution of dimethyl-*p*-phenylenediamine in alcohol, and the mixture is kept for 10—15 min. A red coloration develops, the intensity being proportional to the chlorine content; a standard iodine solution treated in the same way is used for a comparison test if quantitative results are required. The test will detect 0·03 mg. of chlorine in 1 litre. S. I. LEVY.

Natural and artificial filtration [of water] in connexion with goitre. J. DON (Kolloid-Z., 1926, 38, 334—335).—A discussion of the connexion of goitre with the iodine content of Dutch waters. N. H. HARTSHORNE.

Experiments on the purification of sewage water on aero-filters in 1923 [in Moscow]. (Miss) N. A. BAZIAKINA (Moscow Communal Management: 5th Rept. of the Committee for the Purification of Sewage (Russ.), 1925, 1, 127—136).—The results are given of a series of experiments on the purification of Moscow sewage waters by means of two aero-filters, each 3·5 m. in height and charged with slag of 10—25 mm. size in one case and 2—10 mm. in the other. The water, which first passed through a decantation tank, was free from nitrates and dissolved oxygen, had an oxidisability number of 65·0 (by the Kubel method; acid solution, 10 min. at 100°), and contained 109·6 mg. of chlorine and 69·0 mg. of ammoniacal nitrogen per litre. Under the conditions employed, the output per day of the aero-filters was normally 15 cub. m. per day per sq. m. and rose as high as 20 cub. m.; this output greatly exceeds that of percolation beds. The volume of air necessary for the normal functioning of the purification is from 4 to 6 times that of the water treated, the pressure in the air-pipes being from 4 to 20 mm. of water. With the finer slag the duration of the purification is about 25 min., and with the coarser slag about 12 min. T. H. POPE.

Velocity of dissolution of oxygen as one of the factors of biological [sewage] purification. (Miss) N. A. BAZIAKINA (Moscow Communal Management: 5th Report of the Committee for the Purification of Sewage (Russ.), 1925, 1, 139—159).—Since biological oxidation of sewage water is effected at the expense of the dissolved oxygen, increase in the rate at which the oxygen dissolves, which in aeration tanks and bacteria beds is slow, would accelerate the purification. The rate at which oxygen is dissolved is proportional to its deficiency, so that $dx/dt = k\ (b—x)$, where b is the amount of oxygen in solution saturated with air, x is the amount actually present at any time, and k is a constant. The value of $(b—x)$ depends on the speed with which the activated sludge transmits the oxygen for the oxidation, and k depends on the intensity of the aeration, the method of distributing the air, the temperature, etc. With percolation beds the ventilation is defective and the partial pressure of the oxygen consequently low. In aeration tanks, with distribution of the air through perforated pipes, the surface of contact between the air and the liquid is considerably increased, whilst in aero-filters there is perfect combination of the activated sludge, the sewage water, and the air, resulting in maximum velocity of dissolution of the oxygen and hence of the purification process. Aeration tanks, on the other hand, allow of greater variation in the duration of the purification, and are hence better adapted to yield effluents of any desired quality. T. H. POPE.

Present state of the treatment of sewage by means of activated sludge. S. N. STROGANOV

(Moscow Communal Management: 5th Report of the Committee for the Purification of Sewage (Russ.), 1925, **1**, 177—304).—A historical and critical survey of this question. T. H. POPE.

Bactericidal action of nitroso-compounds. E. A. COOPER and G. E. FORSTNER (J.S.C.I., 1926, **45**, 94—96 T).—The group of organisms comprising *Bacillus pyocyaneus*, *B. fluorescens liquefaciens* and *non-liquefaciens* are more susceptible than *B. coli* to those disinfectants, *e.g.*, phenol, the bactericidal action of which appears to be due to their interference with the colloidal condition of the bacterial protoplasm, and which are therefore termed "physico-chemical" germicides. On the other hand, those disinfectants which react chemically with the protoplasmic constituents selectively attack *B. coli*, and the *B. pyocyaneus* group are extremely resistant to their action. From bacteriological results it is concluded that nitroso-compounds belong to the second class of bactericidal substances. Chemical evidence for the foregoing biological differentiation has been obtained by a study of the action of nitroso-compounds on proteins. It would appear that these compounds react chemically with the cell-globulins and proteoses, and this reactivity may account for their high germicidal power. An iodometric method for determining nitrosophenol is described.

Bactericidal action of the *cyclo*telluropentanediones and their derivatives. G. T. MORGAN, E. A. COOPER, and A. E. RAWSON (J.S.C.I., 1926, **45**, 107—109 T).—The *cyclo*telluropentanediones are much more active germicides than various other tellurium derivatives that have been examined, and bactericidal power in general bears no relation to the percentage of the metalloid present in the substance. The *cyclo*telluropentanediones are also considerably more efficacious than the corresponding oximes. The outstanding bactericidal power of the foregoing germicides disappears in the presence of serum, and this collapse in activity has been shown to be due to the presence of small amounts of fats and fatty acids, which apparently adsorb the tellurium compounds from dilute solution and thus render them unavailable for disinfection.

Effectiveness of certain paraffin derivatives in attracting flies. W. C. COOK (J. Agric. Res., 1926, **32**, 347—358).—A number of aliphatic alcohols and esters have been tested in regard to their attraction for flies over a uniform period of exposure of 24 hrs. The most attractive concentration bears a close relation to the boiling point of the compound, decreasing rapidly with a rise in boiling point. Further, the relative attractiveness of these compounds decreases with rise of boiling point. The addition of a CH_2 group to the acid radical reduces the attractiveness much more than addition of the same group to the alcohol radical. *iso*-Compounds are relatively more attractive than their normal isomerides. C. T. GIMINGHAM.

Corrosion of iron pipes. BAYLIS.—See X.

Fungicidal properties of spray fluids. GOODWIN, MARTIN, and SALMON.—See XVI.

Toxicological investigations on mandibulate insects. CAMPBELL.—See XVI.

PATENTS.

Purifying of boiler sludge and the heating and supply of boiler feed water. NECKAR WATERREINIGER MAATSCHAPPIJ, and J. HERINGA (E.P. 250,061, 16.6.25).—The invention comprises a combination of a heater, purifier, and tank for feed water and an expansion tank for blow-down water from a boiler. The sludgy water from a boiler is blown off into the expansion tank, the steam from which is mixed with and heats the purified feed water and at the same time provides an atmosphere of steam in the tank, preventing solution of gases from the atmosphere. The separated sludgy water, reduced to a temperature of about 100°, if pure (except for suspended matter) or containing useful excess purifying agents from a previous treatment, passes to the feed water purifier where it mingles with raw feed water. If, on the other hand, the water contains dissolved harmful salts such as sodium chloride or sulphate, part or all of it passes through an indirect heater for the feed water and is then discarded. B. M. VENABLES.

Softening water. Water-softening material. R. G. TELLIER, Assr. to PERMUTIT CO. (U.S.P. 1,571,891—2, 2.2.26. Appl., [A] 27.5.16, [B] 6.9.21). —Hard water is passed through a granular bed of base-exchanging material prepared from a natural clay. Clay containing a substantial proportion of hydrated silicate of a zeolitic nature is cut into small pieces, immersed in a hot solution of sodium chloride, dried at ordinary temperatures, baked at about 110°, granulated, then (A) heated below 700° for $\frac{2}{3}$—$1\frac{1}{2}$ hrs. and finally treated with a hot solution of sodium chloride, or (B) heated to temperatures above 700° and re-hydrated by immersion in a hot solution of caustic soda. W. T. LOCKETT.

Water purifier. A. L. GRANT (U.S.P. 1,574,477, 23.2.26. Appl., 13.12.23).—The permanent hardness of water is removed by treatment with finely divided barium silicate, preferably in the presence of magnesium carbonate (contained in the water or added thereto), sufficient in quantity to satisfy the equation, $CaSO_4+MgCO_3+BaSiO_3=MgSiO_3+CaCO_3+BaSO_4$. W. T. LOCKETT.

Base-exchange water softener. O. R. SWEENEY (E.P. 244,303, 7.3.25).—See U.S.P. 1,557,117; B., 1926, 110.

Obtaining boiler feed water by distillation. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (E.P. 247,567, 10.2.26. Conv., 10.2.25).

Preparation of sterilising agents, germicides, and the like. C. H. H. HAROLD, Assr. to UNITED WATER SOFTENERS, LTD. (U.S.P. 1,581,115, 20.4.26. Appl., 3.10.25).—See E.P. 245,820; B., 1926, 302.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 9, 1926.

I.—GENERAL; PLANT; MACHINERY.

Influence of rate of stirring on reaction velocity. F. C. Huber and E. E. Reid (Ind. Eng. Chem., 1926, 18, 535—537).—The rate of reaction is approximately a linear function of the rate of stirring in reactions such as the ethylation of benzene by ethylene, the oxidation of sodium arsenite by oxygen, etc., where the reaction of the dissolved gas is rapid but the solution of the gas is slow, and therefore affected by the stirring. Reactions such as the catalytic hydrogenation of cottonseed oil or a hydrocarbon are approximately linear functions of the rate of stirring when a certain rate has been reached; in this case, a certain rate of stirring is necessary to ensure uniform distribution of the catalyst and thus promote hydrogenation. Reactions such as the saponification of ethyl benzoate or the hydrolysis of benzyl chloride not affected by the rate of stirring.

B. W. Clarke.

Patents.

Furnaces for heating at low temperatures. F. Puening (E.P. 228,148, 14.1.25. Conv., 23.1.24). —A large quantity of gas is reciprocated over the goods to be heated by an apparatus comprising a water-sealed bell moving up and down within a similarly shaped container; at each stroke the reciprocating gas acquires slight increments of heat by admixture with products of combustion, similar amounts of excess gas being exhausted to atmosphere. One bell-piston may serve several heating chambers, in which case the stroke of the bell is lessened when heating chambers are cut out for repairs.

B. M. Venables.

Kiln. H. F. Gruetzemacher, jun., Assr. to Glencoe Lime & Cement Co. (U.S.P. 1,580,483, 13.4.26. Appl., 8.10.23).—A combustion chamber adjacent to the lower part of the kiln is provided with means for supplying fuel and air thereto and for preheating the air. The air is passed through a heating chamber within the walls of the kiln above the combustion chamber and thence into the combustion chamber through a conduit external to the kiln. Means are provided for controlling the velocity of circulation of the air and for otherwise controlling its temperature.

H. Holmes.

Refractory brick for furnace linings. O. Aram (U.S.P. 1,576,021, 9.3.26. Appl., 14.9.22).—A surface coating, consisting largely of metallic aluminium, is applied to exposed surfaces of the refractory lining, the durability of which is thereby prolonged.

F. Salt.

Absorption refrigerating apparatus. Siemens-Schuckertwerke G.m.b.H. (E.P. 229,332, 13.2.25. Conv., 14.2.24).—In an absorption refrigerating system the difference in pressure between the generator side and the absorption side of the apparatus is utilised by means of an injector to cause movement of the gas in the low-pressure side from the evaporator to the absorber. This permits a wider choice of inert carrier gas; for example, air at about atmospheric pressure may be used for this purpose, with the advantage that small leaks are comparatively harmless.

B. M. Venables.

Evaporating process and apparatus. Soc. Gén. d'Evaporation Proc. Prache & Bouillon (E.P. 242,259, 16.10.25. Conv., 31.10.24).—The liquid to be evaporated is heated and evaporated by passing counter-current in contact with a stream of cold air, the bulk of the liquor being returned through an indirect heat interchanger and preheater to the evaporator. Fresh make-up liquor is added to the return circuit, and the liquor when sufficiently strong is drawn off from the bottom of the evaporator. The warm moist air from the evaporator is propelled by a fan to another contact apparatus where it gives up its sensible and latent heat to a counter-current stream of cold water, the air returning to the evaporator and excess water being drawn off. The heat thus given to the water is transferred back again to the liquor in the above-mentioned heat interchanger, and additional heat to compensate for losses and to provide for the temperature drops is given to the liquor in the preheater, which is heated by live steam or other external means. In the case of liquors which do not deposit crystals the evaporator may consist of a contact tower filled with coke or other filling; if crystals are deposited during evaporation the air may be passed over the surface of the liquor in a vat; and if the material to be evaporated is pasty, the air may be passed through the interior of a rotating drum, the exterior surface of which picks up the material to be evaporated or dried. To transfer the heat of condensation back to the liquor, instead of a simple water circuit, apparatus similar to a refrigerating apparatus, comprising vaporiser, compressor, and condenser, may be used, the necessary external energy being supplied as power to the compressor.

B. M. Venables.

Apparatus for heating and boiling liquids. Blair, Campbell & McLean, Ltd., and R. Webster (E.P. 250,762, 25.4.25).—In apparatus for heating liquids by steam, comprising a number of units of twin concentric pipes forming annular chambers for

the steam, the units are arranged to be held together and to the steam supply header solely by means of a nut on the inner tube. B. M. VENABLES.

Multiple-effect evaporative separation. H. K. MOORE, Assr. to BROWN Co (U.S.P. 1,582,066–7, 27.4.26. Appl., [A] 16.10.22, [B] 30.11.23).—(A) In a multiple-effect evaporating process the liquor flows from an intermediate effect through progressively hotter effects and then through effects that are progressively cooler. (B) Liquor which would become viscous if completely concentrated in the cooler effects, is supplied hot to a heat interchanger and cooled to the temperature of an intermediate effect and is passed through that and a series of cooler effects. From the coolest effect the liquor is reheated to the temperature of the effect next hotter than the starting point by means of the heat interchanger, and then passes through a series of hotter effects, being heated while passing to each of these effects by heat taken from the liquor leaving the hottest effect of the series. B. M. VENABLES.

Evaporator. J. A. PAUL (U.S.P. 1,580,323, 13.4.26. Appl., 29.4.21).—The open end of a cylindrical evaporator drum is closed by a cylindrical curved spring metal cover in such a way that only the edges are adapted to yield when the internal pressure exceeds a predetermined amount. H. HOLMES.

Heat treatment of materials for the extraction of volatile constituents or moisture therefrom. W. R. HUME (E.P. 250,302, 29.12.24).—The material is fed to the interior of a rotating drum against the walls of which it is held by centrifugal force. The drum is subjected to external heat and if desired is also heated internally by steam or electricity. Various devices for feed inlets, product outlets, and liquid seals are described. B. M. VENABLES.

Rotary drums for cooling or otherwise treating materials by air currents. J. S. FASTING (E.P. 250,318, 8.1.25).—A rotary drum, *e.g.*, a cooler connected to a rotary cement kiln, is provided with metal cylinders, rings, bands, plates, or the like, having a large surface relative to their weight, connected to the inner surface of the drum by chains permitting a limited amount of free movement, and arranged within the drum to attain the maximum amount of transference of heat from air to the material or *vice versa.* B. W. CLARKE.

Measurement of [flow of] heat. A. BLACKIE (E.P. 250,416, 29.5.25).—The apparatus comprises a heat-receiving vessel containing air or other fluid connected to a capillary orifice and a pressure gauge or manometer. The rate of flow of heat into the vessel is calculated from the reading of the manometer when equilibrium is reached between the expansion of the air and its outflow through the orifice. The face of the vessel which receives the heat is preferably blackened and the other faces may be protected by vacuum or water jackets. The vessel itself may form its own pressure gauge on the aneroid principle. B. M. VENABLES.

Tubular mills for grinding or crushing. O. BOUZIN (E.P. 250,504, 28.12.25).—A grinding mill of the type where a number of separate cylindrical grinding chambers rotate about a common axis, is provided with feed and outlet chambers common to all the cylinders, the feed chamber being provided with a scoop for each cylinder. B. M. VENABLES.

[Gyratory] crushers. ALLIS-CHALMERS MANUF. CO., and R. C. NEWHOUSE (E.P. 250,749, 3.4.25).—In a gyratory crusher of the type which has a conical crushing head and shaft, depending from a universal bearing at the top and oscillated by an eccentric at the bottom, the eccentric is driven at high speed direct from a motor at the top of the apparatus, through a rotating shaft within the hollow gyrating shaft. The gyrations are so rapid that the pieces of material being crushed never have time to come to rest. To prevent harmful effects of vibrations the whole crusher and motor is suspended freely by means of three ropes. B. M. VENABLES.

Pulveriser mill. A. KELLER (U.S.P. 1,580,610, 13.4.26. Appl., 25.1.24).—A grinding ball within a grinding ring co-acts with a pusher member having a constantly shifting and rolling contact with the ball. H. HOLMES.

Pulveriser. T. L. MCKAIN (U.S.P. 1,580,620, 13.4.26. Appl., 24.5.22).—A cylindrical drum is provided with rotary beaters mounted axially and extending close to but not in contact with its inner surface. The lower part of the lower half of this surface is perforated, the remaining portions of the lower half are smooth and non-perforated, and the whole of the upper half is corrugated to provide longitudinal ribs. H. HOLMES.

Pulverising mill. F. I. RAYMOND, Assr. to RAYMOND BROS. IMPACT PULVERIZER CO. (U.S.P. 1,582,538, 27.4.26. Appl. 25.6.23).—A pulverising mill of the ring and roller or similar type is provided with an inlet for material above the grinding zone, and air is admitted below the grinding zone in such a manner that it first moves outwards and then upwards and inwards, passing through the grinding zone and separating the material as it falls therefrom. B. M. VENABLES.

Pulverising mill. C. B. STRACHAN, Assr. to STRACHAN TUBE MILL CO. (U.S.P. 1,583,324, 4.5.26. Appl., 25.5.25).—The grinding elements comprise a number of cylinders of different diameter assembled loosely and coaxially within a rotating cylindrical casing. Cylindrical spaces for entry and exit of material are left between the ends of the mill and perforated inner diaphragms which serve to retain the grinding cylinders in place. B. M. VENABLES.

Carrying out chemical reactions. E. BUHTZ (E.P. 250,722, 26.2.25).—The substances which are to react together are liquefied and supplied, preferably through concentric conduits, to a substantially horizontal disc rotating at a low speed. The disc may be dished or funnel-shaped, may be provided with corrugations or rings to increase the surface and

provide strong agitation, and may work in conjunction with a smaller disc which may also rotate.
B. M. Venables.

Controlling operations of solvent-recovery plants. G. G. Oberfell (U.S.P. 1,580,157, 13.4.26. Appl., 18.4.25).—The vapour pressure of a liquid mixture is determined by bringing a definite volume of air into contact with the mixture and measuring the increase of volume of the air due to its saturation with the vapour. H. Holmes.

Drying apparatus. G. J. Pezold (U.S.P. 1,580,164, 13.4.26. Appl., 28.1.24).—The cylindrical wall of a rotary drum is provided on its opposite ends with flanges projecting inwards, and hollow trunnions are arranged on heads secured to the flanges. An inner shell, spaced from the wall, is secured at its opposite ends to the flanges, and the space between the shell and the wall constitutes a heating chamber. Transverse partitions, spaced from the heads, are secured within the shell, and the space between one partition and the adjacent head forms a conduit for the heating fluid which passes into the heating chamber through perforations in the adjacent part of the shell. H. Holmes.

Rotary drying apparatus. C. Miller (U.S.P. 1,581,359, 20.4.26. Appl., 7.3.25).—A rotating vertical cylindrical container is supplied with air from a fan at the bottom, the stream of air being divided by a perforated plate into several currents. The air is exhausted through several outlets at the top. B. M. Venables.

Anti-freezing solution. E. H. Crofoot, Assr. to Larrowe Construction Co. (U.S.P. 1,581,179, 20.4.26. Appl., 16.1.20. Renewed 18.9.25).—An anti-freezing solution for radiators consists of the waste liquor from sugar extraction processes.
A. R. Powell.

Separation of liquid mixtures by distillation. E. Merck (E.P. 244,036, 29.4.25. Conv., 8.12.24). —See F.P. 595,821; B., 1926, 176.

Annular furnace with revolving hearth for continuous working. L. Honigmann (U.S.P. 1,583,974, 11.5.26. Appl., 20.5.24).—See E.P. 237,376; B., 1925, 742.

Separation of the solid constituents of liquids containing them. H. N. McLeod (U.S.P. 1,585,300, 18.5.26. Appl., 28.6.24).—See E.P. 248,081; B., 1926, 392.

Means for reducing air currents in centrifugal machines. K. J. Svensson and K. A. P. Norling (U.S.P. 1,584,488, 11.5.26. Appl., 3.8.23).—See E.P. 202,616; B., 1924, 736.

Drying process and apparatus. T. Rigby (U.S.P. 1,585,511, 18.5.26. Appl., 13.2.22).—See E.P. 182,157; B., 1922, 800 A.

Manufacture of porous masses for storing gases. Gas Accumulator Co. (United Kingdom), Ltd. From Autogen Gasaccumulator A.-G. (E.P. 242,241, 8.9.25).—See G.P. 420,150; B., 1926, 230.

Means for supplying heated air to furnaces and the like. A. Crowcroft (E.P. 251,318, 14.8.25).

[Regulating supply of air to] furnaces. E. Roucka (E.P. 228,924, 6.2.25. Conv., 7.2.24).

Heat exchange apparatus. R. Seligman (E.P. 251,064, 13.3.25).

[Device for cleaning] centrifugal separators. De Laval Chadburn Co., Ltd., and A. C. Bamford (E.P. 251,738, 18.3.25).

Vacuum-producing apparatus. [Water-jet gas ejector.] G. Sokolow-Wichnevsky (E.P. 251,858, 12.10.25).

Centrifugal separator (E.P. 250,319).—See II.

Crucible furnaces (E.P. 238,562).—See VIII.

Electrical gas cleaning (E.P. 250,499).—See XI.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Dependence of the form of the flame in a combustion flue on various factors. P. Stoller (Gas- u. Wasserf., 1926, 69, 374 378).—The influence of (1) form of air nozzle, (2) increased draught, (3) gas velocity and form of burner orifice, and (4) amount of excess air on the form of flame in a vertical combustion chamber, 3·7 m. high and 0·1 m.^{2} cross-section, has been studied by varying one factor at a time and determining the temperature distribution in the chamber and regenerator. A rectangular air nozzle gives a more regular temperature distribution than a "whirling" nozzle, but a somewhat lower temperature in the combustion chamber and a higher temperature in the regenerator. Increased draught lowers the maximum temperature and moves its position nearer to the base of the chamber. A more regular temperature distribution is attained by varying the cross-section of the gas and air apertures so that the gas velocity and air velocity are nearly the same. At the same time a rise of temperature occurs in the regenerator, and the efficiency of the combustion is lowered. With increasing excess of air the height of the flame decreases. A. B. Manning.

Gases and the carbonisation of fuels. P. Lebeau (Bull. Soc. chim. Belg., 1926, 35, 47—74). —Extended researches on the gases liberated on the heat treatment of various fuels at temperatures up to 1000° are summarised. Starch, certain sugars, casein, and amygdalin were also examined (cf. B., 1923, 877 A, 964 A; 1924, 206, 585, 700, 775; 1925, 88, 744). W. Clark.

Active charcoal. H. Chaumat (La Nature, 1926, 149—155; Chem. Zentr., 1926, I., 2891).—The absorptive power of charcoal is greater the freer it is from certain hydrogen compounds; it is greater also, when comparing different absorbed substances, the greater the heat effect associated with the absorption. The absorption is not in

accord with Henry's law. Activation is produced by treatment with sulphuric acid, zinc chloride, or phosphoric acid, of which the last is the most efficient. The phosphorus formed on heating the charcoal to 600° with phosphoric acid reacts with the hydrogen compounds, producing hydrogen phosphide. The excess of phosphoric acid is removed by washing, the charcoal dried, and ignited at 1200°; it is then washed with hydrochloric acid and finally dried at 300°. The charcoal so treated contains only 0·2 instead of 4·5% H, and can absorb 65—70% of its weight of benzene.

A. B. Manning.

Determination of the porosity of coke, refractory bricks, and rammed moulding sand. H. Esser and E. Pivovarski (Stahl u. Eisen, 1926, **46**, 565—567).—An apparatus for the rapid determination of the true and apparent volume of a porous body is described. The principle of the method is similar to that of Washburn and Bunting (B., 1922, 710 A; cf. Pressler, B., 1924, 381). The air in a glass container is rarefied by sucking a portion into a measuring burette, and after introducing the sample the operation is repeated to the same diminished pressure in the container. The difference between the quantities of air measured in the burette in the two operations multiplied by the quotient, barometric pressure/pressure in container after withdrawal of air, gives the volume of the substance. The apparent volume is found in the same apparatus by the mercury displacement method. From these figures the porosity is calculated as usual.

A. R. Powell.

Liquid purification of coal gas and the recovery of sulphur. C. Harnist (Chim. et Ind., 1926, **15**, 506—513; cf. Berthelot, B., 1926, 36).—The American, Claus, Burkheiser, Feld, and other processes for the purification of coal gas and the recovery of sulphur therefrom are summarised and critically compared. The American process, in which the hydrogen sulphide is absorbed in a solution of sodium carbonate which is regenerated by the passage of a current of air, makes recovery of the sulphur difficult owing to the very low concentration of hydrogen sulphide in the air current. The modification of the process in which the alkaline solution contains suspended ferric oxide, is unsuccessful in practice owing to the difficulty of dealing with the precipitated material. The recovery of sulphur by the Claus process is not yet a commercial success. Although the Feld process, in which the hydrogen sulphide and ammonium sulphide are oxidised by sulphur dioxide to a mixture of polythionates and sulphur, the polythionates being subsequently converted into ammonium sulphate, has yet to overcome many practical difficulties, it appears to be the most promising as a basis for future developments.

A. B. Manning.

Determination of paraffin hydrocarbons. W. Steuer (Gas- u. Wasserf., 1926, **69**, 417—419).—An improved method of determining methane and ethane consists in burning the hydrocarbons mixed with oxygen over red-hot platinum or palladium. The methane–ethane mixture is measured in one Hempel burette, the oxygen in a second. The two burettes are connected by 10 cm. of transparent quartz capillary tubing containing 3 cm. of platinum or palladium wire. The gases are mixed by passing the oxygen into the other burette, the quartz tube and contained catalyst are heated by a full Bunsen flame, and the mixture is passed over the catalyst slowly in order to avoid explosion. The catalyst is then raised to a bright red heat, and the gas mixture passed over it repeatedly until combustion is complete. The amounts of methane and ethane are calculated from the total contraction and the carbon dioxide formed. Palladium induces combination more rapidly and at a lower temperature than platinum. Ethane burns at a lower temperature than methane. For this method, rapidity, accuracy, and facility of operation are claimed.

R. A. A. Taylor.

Ultra-violet spectroscopy of flames of motor fuels. G. L. Clark and W. C. Thee (Ind. Eng. Chem., 1926, **18**, 528—531).—The spectra of detonation flames in an internal-combustion engine give a characteristic banded structure, readily distinguishable from the spectra of explosion and combustion. Lead tetraethyl causes some dispersion of the banded spectrum and a shortening of the ultra-violet region; butyl nitrite (a detonation inducer) accentuates the ultra-violet spectrum. Characteristic lead lines are prominent when lead tetraethyl is used, and show that the process of decomposition of the lead tetraethyl molecule is the important factor, the combination with bromine introduced as ethyl bromide taking place subsequently. The persistence of these lead lines with only minute traces of lead tetraethyl present affords the most delicate test for the presence of this compound. Copper lines from the plug spark appear as emission lines when lead is present in the vapour, but as absorption lines in its absence. The study of the spectra of the flames in the internal-combustion engine promises to be a satisfactory method of elucidating the reaction mechanisms under varying conditions.

B. W. Clarke.

Wurtemburg oil shales. II. F. C. Gaisser and H. Bader (Chem.-Ztg., 1926, **50**, 277—280).—A rapid method of extracting the organic matter from oil shale consists in stirring it into 10—15 times its weight of fused sodium hydroxide, heating for 5—10 min., extracting the fused mass with water, and treating the insoluble material with hydrochloric acid. The organic residue is collected and dried at 105° for analysis in the usual way. From Wurtemburg oil-shale the product so obtained consisted of 19·6% of a voluminous, finely divided, brown powder containing 63·92% C, 7·18% H, 2·15% N, 12·83% O, and 7·4% S; the shale is therefore more of the nature of a coal-shale than of a true oil-shale. Distillation of this type of shale yields 7·8% of tar, 3·3% of water, 85% of "coke," and 3·9% of gas, but if the organic matter is first separated as above and then distilled the yield of tar is 8·5% and that of coke 8·3% (containing 22% of ash). The gases

obtained in the latter operation are rich in hydrogen sulphide and the tar contains a large proportion of sulphur compounds. A. R. Powell.

Effect of pressure and temperature on total volume of partially vaporised Midcontinent crude [petroleum]. R. E. Wilson and H. G. Schnetzler (Ind. Eng. Chem., 1926, **18**, 523—525).—Variations in the water content of the crude oil produce large differences in the ratio of volume of vapour+liquid to volume of cold liquid over a wide range of temperatures and pressures. The shape of the curves obtained by plotting the results indicates solubility of water in the oil. At moderate pressures and temperatures above 150°, the volume of vapour produced is so large that it must be allowed for in calculations concerning design of rectifying stills. B. W. Clarke.

Bergin benzine and Bergin oil. E. László (Petroleum, 1926, **22**, 421—425).—A mixture of 100 kg. of Italian lignite with 67 kg. of topped coal tar and 4 kg. of "Lux" material was "berginised" in a 50-litre bomb by the continuous method, with the addition of 5 kg. of hydrogen. The finely-ground lignite was mixed with the tar to form a paste. The lignite contained 13·8% of water, 8·45% of ash, and 0·8% of sulphur. Hydrogenation took place at 470° under a pressure of 140—150 atm. The expansion gases were freed from benzine by activated charcoal, yielding 8 kg. of benzine. 20 kg. of gas were formed and 139 kg. of "runnings," and the loss was 9%. The benzine was water-white, had an odour of petroleum benzine, and *d* 0·7303. 84·6% distilled over from 50° to 120°, and the benzine was easily refined. The relation between b.p. range and *d* corresponded to that of American benzine. The lower fraction obtained by distilling the "runnings" had *d* 0·990 and contained 12·5% of phenols, being similar to a low-temperature tar oil. The wax content was 1% to 1·5%. The residue was very viscous, and on re-distilling 47% of hard asphalt remained, indicating a total asphalt content of about 60%, but the ash content was small. A third fraction of the "runnings" consisted of water (29·5 kg.) containing 7·83 g. of ammonia per litre. This only corresponded to 19% of the total nitrogen present in the raw material, the rest having escaped as ammonia in the expansion gases. H. Moore.

Nature of the protoparaffin waxes in petroleum. A. Sachanen (Sachanov) and M. Bestuschev (Petroleum, 1926, **22**, 484—486).—Protoparaffins were precipiatated by ethyl alcohol from an *iso*amyl alcohol solution of Grosny petroleum, refined with acid, and treated in benzine solution with floridin; 3% of wax with m.p. 51·5—53·5°, and 3% with m.p. 45—47° were obtained. A third sample of protoparaffin wax was obtained from the precipitate from the inner surface of a pipe line. This, after refining, had m.p. 75—77°. The solubility of these samples in light benzine, machine oil, benzene, and *iso*amyl alcohol was investigated, and the results are given in tables and graphs. The protoparaffins dissolve as true crystalline substances, and there is no difference between them and pyroparaffins, observed differences of behaviour being due to the accompanying substances in crude oils and residues. H. Moore.

Adsorption of asphalt from mineral oils or residues by hydrosilicate earths. H. Herbst (Petroleum, 1926, **22**, 424).—1000 g. of residue from kerosene distillation were diluted with 250 g. of kerosene. The mixture had an asphalt content of 8%, and was filtered through a layer of mixed powdered and granular "Silit" 15 cm. thick, weighing 580 g., at 60°. After 48 hours filtrate commenced to pass, and 95 g. were collected in a further 48 hours, being light-yellow and free from asphalt. A second fraction, 65 g. free from asphalt, was collected, and after three days a further 150 g., dark in colour and containing 7% of asphalt. Thus the "Silit" was exhausted after adsorbing only 2·5% of its weight of asphalt. The first fraction of filtrate was of slightly lower density than the succeeding fractions. H. Moore.

Influence of rate of stirring on reaction velocity. Huber and Reid.—See I.

Determination of small quantities of hydrogen in gaseous mixtures. Lebeau and Marmasse.—See VII.

Patents.

Plant for drying fuels, together with installation for electrical precipitation of dust. Siemens-Schuckertwerke G.m.b.H. (G.P. 425,499, 27.11.21).—Propagation of ignition from the precipitator to the drying chamber is prevented by the cooling and baffling action of a fan installed between the two. J. S. G. Thomas.

Burning pulverised fuel. H. Kreisinger, J. Anderson, and J. E. Bell, Assrs. to Combustion Engineering Corp. (U.S.P. 1,581,351, 20.4.26. Appl., 30.12.20).—A stream of pulverised fuel is projected downwards into a combustion chamber from which the outlet for flame is at the top, so that the flame takes a U-shaped course. Air is admitted parallel to, and on each side of, the fuel jet, and an additional slower current of air, drawn in by the natural draught of the furnace, is admitted between the descending and ascending legs of the U in order to control the depth of the U. B. M. Venables.

Carbonisation of coal, peat, wood, and the like. C. B. Winzer and P. A. Brown (E.P. 250,661, 14.1.25).—The material is charged into chambers which move continuously in a cycle in a circular or straight muffle or oven. The charge is heated progressively and the distillation products are drawn off in stages. The carbonised residue finally reaches an unheated zone in the plant where it is cooled and discharged. In the heated zone the heating gases are caused to follow a helical path around the chambers. The charge may be introduced into the chambers in basket containers. R. A. A. Taylor.

Treatment [distillation] of carbonaceous materials. A. M. A. Struben (E.P. 250,699, 26.1.25).—Before distillation the material is mixed

with a substance which has a boiling point higher than that of the least volatile product required to be distilled. A suitable retort for the process consists of a horizontal drum fitted with paddles and other necessary appurtenances, including a condensing system and a still for fractionating the distilled products and recovering the added material.
R. A. A. TAYLOR.

Standpipe apparatus [for carbonisation plant]. J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,581,556, 20.4.26. Appl., 13.12.23).—The top of the ascension pipe from a carbonisation chamber is connected with a discharge pipe which curves over and downwards and leads into a pipe, slightly inclined to the horizontal, which enters the tar main below its longitudinal axis. At the junction of the discharge pipe and the inclined pipe is a valve which moves upwards on to a seating at the end of the discharge, in order to shut off communication between the ascension pipe and the tar main. In the discharge pipe is a spray-nozzle, directed towards the junction of the discharge and inclined pipes, thus playing on the valve. R. A. A. TAYLOR.

Lignite distillation apparatus J. E. NEUMAIER, Assr. to LIGNITE PRESSED COAL Co. (U.S.P. 1,582,297, 27.4.26. Appl., 1.2.22).—An inclined revolving cylinder is mounted on bearings in a head at each end. Heating means are provided and the material is introduced through an inlet in one head, passes via independent passages through the inclined cylinder, and out through the other head. The heads are mounted on trunnions which permit of variation in the inclination of the cylinder.
R. A. A. TAYLOR.

Distilling oven. O. LASCHE and C. GEISSEN, Assrs. to KOHLENVEREDLUNG GES. (U.S.P. 1,582,650, 27.4.26. Appl., 15.11.23).—The oven consists of a vertical cylinder with a transversely corrugated wall. It is heated from the inside and enclosed in an outer jacket, and a layer of material to be distilled is formed on the outside of the corrugated cylinder.
R. A. A. TAYLOR.

Carbonisation, distillation, and gasification of fuels. C. A. A. M. ROUX (F.P. 600,472, 2.10.24).—The central portion of the fuel mass is gasified and the radiant heat of the process utilised to heat the outer portion of the charge.—R. A. A. TAYLOR.

Water-gas from liquid hydrocarbons. M. BRUTZKUS (E.P. 250,289, 10.11.24; cf. E.P. 155,776 and 144,915, B., 1922, 735 A).—The reaction is carried out in the cylinder of a Diesel type engine, in a four-stroke cycle. In the first stroke the cylinder fills with superheated steam, *e.g.*, at 400° and 1 atm., and this is compressed in the second stroke to about 31 atm., the temperature thereby rising to 1200°. At the beginning of the third stroke (expansion) the liquid hydrocarbon, *e.g.*, residue from naphtha distillation, coal tar, etc., is injected into the cylinder together with sufficient air to burn 10—15% of it. During the stroke the remainder of the hydrocarbon reacts with the steam to form water-gas rich in hydrogen, which, during the fourth stroke, escapes through the exhaust into the gas-holder.
A. B. MANNING.

Gaseous fuel. J. HARRIS (U.S.P. 1,581,441, 20.4.26. Appl., 2.6.23).—A mixture of 10—50 pts. of acetylene and 90—50 pts. of coal gas by volume is claimed as a fuel for cutting and welding.
R. A. A. TAYLOR.

Manufacture of methane. SOC. D'ETUDES MINIÈRES ET IND. (F.P. 590,744, 15.2.24).—Mixtures of carbon monoxide and hydrogen, after a preliminary heating, are led over asbestos or pumice coated with rhodium black. A. COULTHARD.

Debenzolisation of gas. COMP. POUR LA FABR. DES COMPTEURS ET MATÉRIAL D'USINES À GAZ (F.P. 596,498, 9.4.25).—The wash oil is allowed to circulate while air is excluded. R. A. A. TAYLOR.

Electric flue gas testing apparatus. G. SOKOLOV-WICHNEVSKY (E.P. 250,478, 12.10.25).—A gas-testing apparatus of the katharometer type is provided with a tubular cooler which at its lower end is connected to a water jet pump, whilst a gas filter and the katharometer cell containing gas and air chambers are arranged at its upper part.
J. S. G. THOMAS.

Liquid fuel. E. M. FLORES (E.P. 249,577, 28.10.24).—Carburetting substances such as crude petroleum or a petroleum fraction 150—220°, or products of the distillation of coal, are added to fermented musts, and, also, if desired denaturing substances and/or a solution of boric acid in methyl and/or ethyl alcohol or acetone, the mixture being kept for a time and distilled once to produce the fuel. The vapours from distillation may be dehydrated by passing over a drying agent, and the distilled fuel may be enriched by treatment with acetylene or other gases. H. MOORE.

Alcohol [motor] fuel. R. DE SARIGNY (E.P. 250,019, 8.4.25).—A fuel suitable for internal-combustion engines consists of alcohol 50%, benzol 12%, petrol 34%, ether 3%, and a denaturant 1%, by volume. The denaturant may consist of pyridine 25% and mineral oil 75%. S. BOWMAN.

Motor fuel oil. F. L. DIETZ (U.S.P. 1,573,307–8, 16.2.26. Appl., [A] 16.11.21, [B] 2.2.22. [B] Renewed 18.7.25).—A fuel for internal-combustion engines consists of (A) low-temperature tar, with the addition of a mixture of alcohol and a hydrocarbon both boiling at 79°, to prevent its solidification; or (B) crude tar from the distillation of coal up to a temperature of 600°, and alcohol. H. MOORE.

[Composition for] prevention of fuel knock. T. MIDGLEY, JUN., Assr. to GEN. MOTORS CORP. (U.S.P. 1,571,862, 2.2.26. Appl., 18.9.23).—The composition consists of aniline, unsaturated gasoline, and a blending agent. A. J. HALL.

Recovering oil from oil-bearing sands. D. C. COLLIER (U.S.P. 1,570,205, 19.1.26. Appl., 8.4.21).

—The material is thoroughly agitated with a mixture of water and crushed carbonaceous matter, *e.g.*, coal. The oil and coal become agglomerated and float on the water while the sand settles out. The mixture of oil and coal may be used as a fuel, or the oil may be separated by distillation.

R. B. Clarke.

Removing water from oils or other liquids. A. Lozai (E.P. 239,833, 9.7.25. Conv., 10.9.24).—In a heat exchanger consisting of vertical tubes with a heating chamber supplied with steam, the tops of the tubes are provided with openings to form weirs. A jacketed head is connected to the heat exchanger and heated by the fluid used in the interior of the latter. A vacuum pump assures the flow of the vapours evolved in the tubes counter to the flow of the liquid film through the tubes. The lower ends of the tubes are obliquely notched to prevent a film forming across them. Additional vertical pipes form a connexion between the head and a receptacle below the exchanger to lower the pressure in the latter and facilitate the downward flow in the pipes. When water is eliminated from oil in this apparatus the steam is drawn off vertically and does not come into contact with the dehydrated oil.

H. Moore.

Separating the phases of emulsions, particularly of petroleum, by means of an electric current. J. H. C. de Brey, Assr. to Naaml. Vennoots. Bataafsche Petroleum Maatsch. (U.S.P. 1,570,209, 19.1.26. Appl., 11.4.24).—The essential feature of the process consists in the application of a pulsating alternating current, the peak voltage of which is at least double the effective voltage. Petroleum can be freed from small quantities of water in this manner.

R. B. Clarke.

Treating [dehydrating] apparatus for crude oil. H. L. Edwards (U.S.P. 1,578,273, 30.3.26. Appl., 29.3.24).—A longitudinal pipe containing heating sections, closed transversely, but traversed by oil tubes longitudinally, serves as a conduit for the oil. Heating fluid is supplied by a pipe to the sections. Intermediate sections are unobstructed and allow settling out of the water, for which outlets are provided.

H. Moore.

Electrical dehydrator [for crude oil]. D. K. Cason, jun. (U.S.P. 1,579,515, 6.4.26. Appl., 16.8.24).—The apparatus consists of a series of conical plates spaced apart at gradually increasing distances from top to bottom. Alternate plates are earthed and the intervening ones connected to a high-tension circuit.

S. Bowman.

Dehydrating oil. H. C. Eddy, Assr. to Petroleum Rectifying Co. of California (U.S.P. 1,580,591, 13.4.26. Appl., 26.8.24).—Water and solid impurities are separated from an oil emulsion by treating it to agglomerate the fine water particles, allowing the liquid to stratify, and removing the driest portion. A wetter portion is passed through water to wash out water and suspended impurities, and the supernatant oil is removed.

H. Moore.

Distilling petroleum oils and other liquids under high vacuum. O. Y. Imray. From Zieley Processes Corp. (E.P. 248,513, 24.12.24).—In a process for the distillation of the heavier fractions of petroleum to produce saleable lubricating oils without subsequent refining, the oil moves downward through inclined conduits in a shallow stream under a pressure of 50 mm. of mercury or less, being subjected to increasing heat. Instantaneous evaporation of the oil and free egress of the vapours prevent cracking or coking in the conduits. A header supplies oil to the conduits, and exit headers are separately connected to separating towers and thence to fractional condensers, jacketed for a cooling medium. The condensers are connected to duplicate receiving tanks, for convenience in withdrawing the product. To facilitate the removal of vapour each conduit is provided with an internal conduit perforated with small holes, which allow the passage of oil or vapour, but tend to break up any froth formed. The ends of the internal conduit may be partially closed by plugs. The vacuum pump is preferably of the ejector type. The vapour outlets throughout the system are of large capacity, and intercommunicate, to promote equalisation of the pressure.

H. Moore.

Cracking liquid or readily fusible organic complexes [tars and heavy oils] by means of an electric arc. C. Longhi (E.P. 248,830, 10.12.24).—To obtain gases rich in acetylene and also carbon black a submerged arc is used and the material acted upon is kept at a relatively low temperature by circulation through a refrigerating device. The materials used are tars and crude and heavy oils generally. The gases produced consist of hydrocarbons of the paraffin series and a large proportion of acetylene. The sulphur and nitrogen present are converted into hydrogen sulphide and hydrocyanic acid and may be separated from the other products, thus allowing the utilisation of substances of high nitrogen or sulphur content. For the generation of the arc mono-phase, two-phase, three-phase, or direct current may be used. The furnace is gas-tight and constructed of metal, and the arc may be continuous or disruptive. The liquid contents of the furnace are circulated through a refrigerator by means of a pump. The carbon or metal electrodes may be hollow for the conveyance of gaseous or liquid substances into the arc zone. The depth of the liquid layer above the arc has a great influence on the proportion of acetylene produced.

H. Moore.

Cracking process [blending]. J. T. Shevlin. From Universal Oil Products Co. (E.P. 249,604, 29.12.24).—An improvement on the Dubbs process (cf. E.P. 160,236; B., 1921, 292 a). To obtain lighter products a heavy hydrocarbon oil is subjected to a cracking temperature in a heating coil, and thence passes to an expansion chamber and to a dephlegmator. The oil and condensate from the

dephlegmator are returned, together with an independent supply of charging stock, to the cracking coil. The pressure in the dephlegmator may be above atmospheric, but below that in the cracking coil or conversion chamber. More than one dephlegmator may be used, the vapours travelling through them in obstructed paths, fresh charging stock being introduced where desired and the non-vaporised residue being returned to the cracking coil. The non-condensable gas may be returned to the cracking zone to assist in the conversion.
H. Moore.

Treatment of hydrocarbons. E. Goltstein (E.P. 249,895, 30.9.24).—Petroleum and similar hydrocarbons are subjected to the action of light of short wave-length such as is obtained by the use of a mercury vapour lamp. The treatment may be applied to either the liquid or vapour phase during distillation, the effect being polymerisation and consequent change in the physical characteristics of the distillates. For example, using a quartz mercury vapour lamp at 60 volts and 6 amp. the viscosity (Engler) of a Russian mineral oil was increased from 6·4 to 7·8 in ½ hr. S. Bowman.

Pyrogenesis of petroleum. E. M. Clark, Assr. to Standard Development Co. (U.S.P. 1,578,802, 30.3.26. Appl., 3.7.19).—Two separate bodies of heavy oil are confined under pressure in a digester. To one heat is applied, and the vapours pass into the second body, which is cooled to promote condensation of the desired products. The oil displaced from the second body is fed to the first body, until the second body becomes of substantially lower boiling point than the first body, conversion being thus effected at a relatively low pressure.
H. Moore.

Apparatus for converting higher boiling-point hydrocarbon oils into lower boiling-point oils. W. M. McComb (U.S.P. 1,579,554, 6.4.26. Appl., 2.3.21).—Conversion of heavy oils into lighter oils is effected by means of a vertical spiral coil, the lower turns of which have an increased inclination to accelerate flow of oil. The coils are grouped for electrical heating. S. Bowman.

Treating [cracking hydrocarbon] oil. G. Egloff (U.S.P. 1,579,601, 6.4.26. Appl., 26.4.17. Renewed 27.9.22).—An externally heated still is provided with means for producing an electric arc within the charge of oil in the still. The oil is thus heated to a cracking temperature and the vapours are passed through a reflux atmospheric condenser, the reflux returning to the still and the uncondensed gases passing to another condenser connected with a receiver. S. Bowman.

Apparatus for distilling hydrocarbons N. F. W. Hazeldine (U.S.P. 1,580,372, 13.4.26. Appl., 9.5.22).—A tank is divided by a horizontal partition into two chambers. An inner shell within the upper chamber provides a jacket for cooling fluid. A riser extends upwards from an opening in the partition and projects through the bottom wall of the inner shell. The oil to be distilled is supplied to the lower chamber, which is provided with a steam coil. Condensed liquid may be withdrawn at a point beneath the outlet of the riser.
H. Moore.

Desulphurising petroleum oils. J. B. Rather, Assr. to Standard Oil Co. of New York (U.S.P. 1,580,531, 13.4.26. Appl., 9.5.23).—Hydrogen sulphide is dissolved in the oil, which is then mixed with a mixture of sodium hydroxide and litharge, or sodium plumbite. Lead sulphide is produced, suspended in the oil, and promotes reaction between the sulphur compounds of the oil and the plumbite solution, rendering the oil sweet to the "doctor test" and non-corrosive. H. Moore.

Centrifugal separating apparatus [for treating mineral oils]. P. P. Harvey and H. J. Holford (E.P. 250,319, 8.1.25).—A centrifugal separator which is specially suitable for oils containing waxy matter is provided with a number of vertical tangential (almost radial) vanes in the lower part of the bowl, and above these, with a number of deflecting plates of dished or conical shape. The material is fed to a space left among the vanes at the centre of the bowl at the bottom and is separated into its constituents while rising between the radial vanes, the circular deflectors then serving to guide the products to appropriate outlets. Any waxy matter is largely deposited on the radial vanes, hence a considerable quantity can accumulate before cleaning is necessary. B. M. Venables.

Penetrating oil and lubricant. H. N. Copthorne, Assr. to Illinois Graphite Co. (U.S.P. 1,580,233, 13.4.26. Appl., 5.6.24).—Deflocculated graphite is suspended in a distillate of $d^{15·5}$ 0·843 obtained by cracking pentadecane at a distilling range of 220—350°. H. Moore.

Treatment of waste products [acid tar] from acid treatment of petroleum oil. R. A. Halloran, W. N. Davis, and G. A. Davidson, Assrs. to Standard Oil Co. (U.S.P. 1,579,607, 6.4.26. Appl., 6.9.23).—The acid tar resulting from the treatment of petroleum with sulphuric acid is heated under a pressure of 50 lb. per sq. in. either with or without the addition of fuel oil. Separation into clean weak acid and an oil suitable for fuel is obtained.
S. Bowman.

Winning of technically valuable products from the alkaline waste products resulting on refining mineral, tar, and shale oils. Sudfelt & Co., Assees. of M. Gelbke (G.P. 426,947, 27.4.23). —The waste products, freed as far as possible from water, are distilled *in vacuo* with superheated steam as long as volatile constituents, especially unsulphonated oils, are evolved. The residue is decomposed with dilute sulphuric acid. The distillates may be used as lubricants, while the products from the residue may serve as emulsifying media in place of soap or Turkey-red oil, and for the hydrolysis of fats. R. A. A. Taylor.

Apparatus for distillation of carbonaceous substances. E. R. Sutcliffe, Assr. to Pure Coal Briquettes, Ltd. (U.S.P. 1,584,487, 11.5.26. Appl., 13.5.18).—See E.P. 166,161; B., 1921, 618 a.

Apparatus for cracking oils. R. C. Holmes and F. T. Manley, Assrs. to Texas Co. (U.S.P. 1,583,973, 11.5.26. Appl., 26.6.19).—See E.P. 160,200; B., 1921, 291 a.

Apparatus for quenching coke. F. Albach (E.P. 246,497, 22.1.26, and 246,508, 23.1.26. Conv., 22 and 24.1.25).

Burners for liquid fuels. J. Y. Johnson. From Badische Anilin- & Soda-Fabr. (E.P. 251,191, 13.11.25).

Production of oil gas. E. C. R. Marks. From Gasifier Co. (E.P. 251,675, 7.1.25).

Treating waste liquors containing cyanogen compounds (E.P. 250,824).—See VII.

Ammonia from ammoniacal liquor (G.P. 426,863).—See VII.

III.—TAR AND TAR PRODUCTS.

Isolation of homogeneous pyridine through the perchlorate. Arndt and Nachtwey.—See A., May, 525.

Patents.

Recovery of phenols and cresols. R. M. Crawford (U.S.P. 1,582,512, 27.4.26. Appl., 16.4.25).—Oil containing phenol and cresols is washed continuously with an alkaline solution containing sufficient alkali to remove the phenol as an alkali phenoxide, and the cresols are subsequently removed by extracting the oil with fresh alkaline solution. L. A. Coles.

Continuous working up of waste acid obtained in the purification of benzol. H. Menzen (G.P. 426,387, 23.3.24).—Apparatus for the continuous treatment of the waste acid with water, sodium hydroxide solution, and oil, comprises a mixing vessel with a separator below it, storage vessels for the waste acid, sodium hydroxide solution, and hot oil, and a condenser delivering into a separator. L. A. Coles.

Cracking tars (E.P. 248,830).—See II.

Treating alkaline waste products from refining tar oils (G.P. 426,947).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Oxidation and constitution of Sulphur Black. U. Perret (Annali Chim. Appl., 1926, **16**, 69—83).—A description of the preparation of the dye, some of its properties, and examination of the self-heating effects, development of acidity, and changes in dyeing properties during isolation in the dry state. Analysis of various samples showed that recent ones were neutral or nearly so, whereas old ones were often considerably acid. The total sodium and sulphur contents were found to vary considerably, and colours having similar dyeing properties and prepared from the same material may have varying compositions. In some lengthy experiments on drying under various conditions, deterioration nearly always took place. When the dye is suspended in 0·5N-sulphuric acid a considerable portion of the sodium in the dye resists the action of the acid, and also old samples are stated to react acid before all the sodium is transformed into sodium sulphate. It is surmised that there may be certain acidic (possibly sulphonic) groups present which are not explained by the usually suggested formulæ for sulphur dyes, the latter being discussed. Three stages of oxidation can be distinguished: (1) during precipitation of the dye (with the formation of leuco-compound) and action of the air on the sodium polysulphide; (2) oxidation of the dye itself with possible transformation of thiol to sulphonic groups; (3) oxidation of the sulphur in the chain to form sulphuric acid. Old samples of the dye were found to be insoluble in sodium sulphide solution and could not be made soluble by re-fusion with polysulphide, as has been stated. Deficiency of polysulphide in preparing the black may lead to the production of sulphites, and not thiosulphates, on air treatment, and give rise to strongly bluish coloured mother liquors, and reasons are given for assuming that the dye is present here as soluble sulphite compounds. S. B. Tallantyre.

Fluorescope [for detecting small quantities of fluorescein]. H. Marcelet (Chim. et Ind., 1926, **15**, 528; Bull. Soc. Chim., 1926, [iv.], **39**, 803—804; Ann. Falsif., 1926, **19**, 296—298).—A simple apparatus is described for determining small quantities of fluorescein in solution by comparing the fluorescence of the solution with that of a standard solution. A beam of light is divided by means of inclined mirrors so that half illuminates one tube and the other half the comparison tube. The tubes containing the solutions are illuminated horizontally and are viewed from above. For very dilute solutions the fluorescein is extracted with ether and so concentrated before comparing with the standard. A. B. Manning.

See also A., May, 508, **Nitration of decahydronaphthalene** (Nametkin and Madaeff-Ssitscheff). 509, **1-Phenylnaphthalene** (Weisz and Woidich). 511, **Action of sulphur on monochloroanilines. Mechanism of aromatic thionation and structure of sulphide dyestuffs** (Hodgson). 512, **Catalytic reduction of α-nitronaphthalene to α-naphthylamine** (Parrett and Lowy). 513, **Colouring matters derived from thiocarbodibenzidine** (Rossi and Cecchetti). 514, **Influence of sulphur on colour of azo dyes** (Palmer and Reid). 515, **Identification of phenols by means of the spectroscope** (Wales and Palkin); **Colour and constitution. Influence of the methylthiol group alone and in conjunction with the methoxyl group** (Hodgson and Handley). 521, **Preparation of quinizarin** (Reynolds and Bigelow). 522, **"Polymethine" dyes and a general dye**

formula as basis of a new generalisation (KÖNIG). 526, **Preparation and properties of methyl*iso*-propylquinoline-yellow** (PHILLIPS and GOSS). 527, **Quinoline Red and related dyes** (SCHEIBE and FISCHER). 530, **Synthesis of phenylrosinduline** (LANTZ and WAHL). 531, **Derivatives of 2-phenylbenzothiazole** (BOGERT and CORBITT); **Polychrome Methylene Blue** (MACNEAL and KILLIAN); **Dyes from the alkaloids of ipecacuanha** (PYMAN; PALKIN and WALES).

Influence of rate of stirring on reaction velocity. HUBER and REID.—See I.

Neocyanine, a new sensitiser for the infra-red. DUNDON, SCHOEN, and BRIGGS.—See XXI.

PATENTS.

Manufacture of diarylamines. O. Y. IMRAY. From SOC. OF CHEM. IND. IN BASLE (E.P. 250,819, 11.9.25).—Nuclear substitution products of diphenylamine are prepared by the action of nuclear substitution products of aromatic hydrocarbons upon alkali metal compounds of aromatic amines, one or both of the compounds containing at least one nuclear substituent in addition to the halogen atom or amino-group. Catalysts, such as copper or its compounds, may be present during the reaction. For example, 140 pts. of sodium are added at 175—180° to 1800 pts. of *o*-toluidine containing 1 pt. of copper powder; after heating to 160—190° until the sodium has disappeared, 750 pts. of *o*-chlorotoluene are added, and the mixture is heated for 24 hrs. at 250—300° in a closed vessel. The *oo'*-ditolylamine obtained is purified by washing with water, followed by fractional distillation *in vacuo*. L. A. COLES.

Mono-oxamic acids of diaminoanthraquinones. BRITISH DYESTUFFS CORP., LTD., H. M. BUNBURY, and R. ROBINSON (E.P. 250,883, 19.12.24).—Compounds of the type, $NH_2.AQ.NH.CO.CO_2H$ (AQ = an anthraquinonyl residue) are obtained by heating mixtures of diaminoanthraquinones, oxalic acid, and water to 105—110°. The products, which contain an amino-group free to take part in condensations and other reactions, *e.g.*, in the preparation of aminodianthrimides, are dark red to violet powders which are thrown out as gels on the addition of mineral acids to their solution in water or in alkalis; on heating to 180—190° they split off carbon dioxide, yielding formamido-derivatives. L. A. COLES.

Preparing 1 : 8-aminonaphtholsulphonic acids. T. GUBELMANN and J. M. TINKER, Assrs. to NEWPORT Co. (U.S.P. 1,573,056, 16.2.26. Appl., 9.5.25).—α-Naphthylaminesulphonic acids containing a sulphonic group in position 8, or their salts, are fused with caustic alkalis in the presence of ammonia, under pressure. Formation of 1 : 8-dihydroxynaphthalenesulphonic acid is thus avoided. For example, for the preparation of S-acid (1 : 8-aminonaphthol-4 sulphonic acid) 500 lb. of caustic soda, 500 lb. of caustic potash, and 400 lb. of water are dissolved by heating in an autoclave, and 1000 lb. of the monosodium salt of α-naphthylamine-4 : 8-disulphonic acid are added. Ammonia gas is introduced up to a pressure of 100—150 lb. and the autoclave is heated to 190—200° for 3—4 hrs. After releasing the ammonia pressure the mass is dissolved in water and acidified in the usual manner.
R. B. CLARKE.

Reduction of organic compounds with hyposulphites. H. BUCHERER (G.P. 423,029, 1.12.22).—Organic compounds insoluble in water are reduced by treatment with sodium hyposulphite and acetic acid, or other aliphatic monocarboxylic acids, such as formic acid or, particularly in the reduction of mono- or dinitronaphthalenes, stearic or oleic acid. Mixtures of the materials are boiled under a reflux condenser until the compounds have dissolved, or until evolution of sulphur dioxide has almost ceased. Examples are given of the production of acetanilide and sulphanilic acid from nitrobenzene; of α-acetamidonaphthalene and a mixture of two aminonaphthalenesulphonic acids (probably the 1 : 4- and 1 : 2-compounds) from α-nitronaphthalene, of 1 : 8-diaminonaphthalenedisulphonic acid and small quantities of perimidine compounds from 1 : 8-dinitronaphthalene; and of a condensation product of equimolecular proportions of acenaphthenequinone and acenaphthenone from acenaphthenequinone.
L. A. COLES.

Preparation of diphenylene sulphide. A. SCHÖNBERG (G.P. 426,476, 17.7.23).—Diphenylsulphoxide is treated with halogen-free substances capable of removing water. For example, diphenylsulphoxide is warmed with phosphorus pentoxide until a reaction commences and then left to itself. After cooling, the melt is extracted with benzene. Diphenylene sulphide remains on distilling off the benzene. The yield is not so good as when sodamide or potassamide is used (cf. A., 1924, i., 39). A. COULTHARD.

Manufacture of trisazo-dyestuffs. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 251,140, 24.6.25).—See U.S.P. 1,565,344; B., 1926, 185.

Colouring-matter paste. BADISCHE ANILIN- & SODA-FABR., Assees. of E. THEOBALD (U.S.P. 1,584,202, 11.5.26. Appl., 8.6.25).—See E.P. 247,052; B., 1926, 355.

Dyes (F.P. 599,566).—See VI.

Azo dyes (F.P. 600,106).—See VI.

Dyeing artificial silks (G.P. 423,601).—See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemical analysis of cotton. XI. Absorption of Methylene Blue from buffered solutions. D. A. CLIBBENS and A. GEAKE (J. Text. Inst., 1926, **17**, T 127—144).—In a previous paper (B., 1923, 1066 A) it was shown that a measurement of the absorption of Methylene Blue by cotton is profoundly influenced by the presence of traces of acids or alkalis. This is now ascribed to a hydrogen-ion

effect and the test, for analytical purposes, is simplified by having a phosphate mixture in the dye solution so that neutrality (p_H 7) is maintained automatically. The effect of hydrogen-ion concentration has been studied particularly with modified celluloses. The absorption by the oxycelluloses (A., 1925, i., 234; B., 1925, 165) falls rapidly as the acidity of the mixture increases. The product obtained by drying sulphuric acid into cotton also absorbs Methylene Blue freely, but in this instance the absorption does not fall as acidity increases. Using a buffered solution for the oxycelluloses and a solution containing free acetic acid for the other product it is thus possible to distinguish between two common types of cellulose modification which may be encountered in bleaching, even when present in the same material. Some experiences of the quinhydrone electrode for determining hydrogen-ion concentrations in Methylene Blue solutions are described.

Experiments on the absorption of Methyl Green and Malachite Green by caustic-scoured cotton and the effect of the alkali present (recorded as ash alkalinity) are also described. With these dyes it was obvious that much of the apparent absorption was due to precipitation of the colourless dye bases by the free alkali present. When the ash alkalinity was reduced to a minimum by washing with acid the absorptions fell into line with those recorded for Methylene Blue, diminishing as the severity of the scouring treatment increased. J. C. Withers.

Chemical analysis of cotton. XII. Hydrocellulose. (Miss) C. Birtwell, D. A. Clibbens, and A. Geake (J. Text. Inst., 1926, 17, T 145—170).—" Hydrocellulose " is defined as any preparation of cotton cellulose (other than esters) the properties of which have been altered to a greater or less extent by the action of acids, but the work described is limited to preparations which still retain the structure of the cotton hair. Such materials have been obtained by steeping cotton in various acids at temperatures between 20° and 100° (" acid steeping ") and by drying fabric impregnated with small quantities of dilute acids (" acid drying "). Tensile strength, viscosity (in cuprammonium hydroxide solution), copper number, and Methylene Blue absorption measurements have been made on the products in order to explore the effects of temperature, time, and acid concentration. Simple relations between loss of strength, viscosity, and copper number, taken in pairs, are found to hold between wide limits, at any rate over the range of acid attack likely to occur in technical processes. The relation between loss of strength and viscosity leads to a suggestion that for textile purposes the minimum permissible viscosity of a 2% solution of a cotton material in the cuprammonium solvent shall be 1000 times that of water (or log $\eta=1$). Viscosity is most sensitive as an indication of tendering in the early stages, up to the first 20% loss in breaking load, whilst copper number is most sensitive when the material is approaching disruption. A connexion between copper number and viscosity is best brought out by considering the viscosity of the 2% cellulose solution, η, relative to that of the solvent, η_0, this being 0·0152 poise. This gives a new term, log η/η_0, designated V, which is connected with copper number, N_{Cu}, for the hydrocelluloses obtained by acid steeping or by acid drying below 70°, in the expression $N_{Cu}V^2=2{\cdot}6$. This relation is most useful in distinguishing hydrocelluloses from certain oxycelluloses which resemble them closely in other characteristics. The value 2·6 is fairly constant for hydrocelluloses over the important range represented by a loss in breaking load of 15—75%, but for oxycelluloses the figure is distinctly higher and is not constant. When cotton is attacked by sulphuric acid of increasing concentrations it first suffers a loss of affinity for basic dyes, which is the normal effect of acid attack, but then acquires enhanced affinity. In this respect, therefore, the sulphuric acid product resembles oxycelluloses, but it can be distinguished from these because it absorbs Methylene Blue quite as well from an acid solution as from a neutral one (cf. preceding abstract). The product contains sulphuric acid in a state of combination which is not destroyed by the most drastic alkali boiling. The normal effects of alkali boiling on the hydrocelluloses are loss of weight and considerable drop in copper number, but the material does not become much weaker (unless the loss in weight is great) and the viscosity remains constant. The factors governing the rate of attack have also been explored. The connexion between time and copper number, concentration and temperature being constant, is well shown by the expression $N_{Cu}=K.T^{0\cdot6}$. Hydrochloric and sulphuric acids in equimolar solutions attack cotton at about the same speed. A 10° rise in temperature, over the range 20—100°, increases the copper number about 2·3 times. The effect of hydrochloric acid is greatly accelerated by the presence of much neutral salt, an important effect which must be considered when discussing the action of concentrated solutions of zinc or magnesium chlorides on cotton. J. C. Withers.

Chemical analysis of cotton. XIII. Scouring losses. R. G. Fargher and (Miss) L. Higginbotham (J. Text. Inst., 1926, 17, T 233—246).—Using an experimental kier which was found to reproduce technical conditions satisfactorily, a study has been made of the caustic boil under the extreme range of conditions represented by normal works practice. Typical American, Egyptian, and Indian yarns were tested, and the factors determined were scouring loss, residual wax content, and the final Methylene-Blue absorption and copper number. Various modifications of the water, soda, and lime boils have also been tested on the same yarns. The results are interpreted as indicating the relative efficiencies of pressure, alkali concentration, lime, etc. in scouring and of Methylene-Blue absorption and copper number as control tests. In addition, scouring losses during the normal pressure boil with caustic soda have been determined for a wide range of cottons, the literature on this subject containing so many contradictions. The losses ranged from 6 to 9%, American varieties coming at the lower

limit, Egyptian intermediate, and Indian high. Only one cotton was found outside this range, namely Pima, with a loss of 11·5%, but this variety is abnormal in other respects. About half the material lost cannot be accounted for as wax, nitrogenous material, mineral matter, or other substances removable by cold water or cold dilute acids. The above losses in the normal caustic boil do not represent all the material in cotton which is less resistant than "normal" cellulose to caustic alkali ; a further loss of 1—2% occurs if 3% sodium hydroxide is used at 40 lb. excess pressure instead of 1% at 20 lb. J. C. WITHERS.

Literature of keratin (the principal constituent of wool). J. BARRITT (J. Text. Inst., 1926, 17, T 111—126).—A review dealing with the analysis, constitution, reactions, degradation by hydrolysis and by mild oxidation, uses, and properties of keratin, with bibliography.
J. C. WITHERS.

Rancidity and oxidation of fatty oils in regard to wool lubrication. W. RHYS-DAVIES (J. Text. Inst., 1926, 17, T 220—232).—Technical defects caused by the oxidation of wool oils are discussed and traced mainly to the amount of linoleic acid glyceride in commercial olive oils. The Mackey tester is more trustworthy for judging wool oils than such characteristics as iodine value.
J. C. WITHERS.

Detection and determination of glycerol in cotton cloths and sized yarns. G. SMITH (J. Text. Inst., 1926, 17, T 187—191).—For detection, the material is extracted with alcohol and, after removing fat and zinc and magnesium salts present, the glycerol is benzoylated by the Schotten–Baumann method. The dibenzoate has m.p. 72—73°. The less specific colour tests due to Denigès are also described. Provided that more than 1% is present, the "I.S.M." acetin process is satisfactory for quantitative determinations. J. C. WITHERS.

Effect of sizes on the elastic behaviour of flax yarns. J. A. MATTHEW (J. Text. Inst., 1926, 17, T 192—205).—Load-extension diagrams of sized flax warp yarns show that the ordinary sizing process has the effect of reducing the extensibility of the yarn by about half. Very little of this effect is due to an adhesive action of the starch, since, under the conditions on the machine, cold water has the same result. The effect is caused by applying tension while the yarn is wet. J. C. WITHERS.

Effects of humidity on the properties of fabrics, with special reference to the control of humidity during strength tests. R. G. PARKER and D. N. JACKMAN (J.S.C.I., 1926, 45, 47—54 T).—Cotton, linen, wool (ordinary and chlorinated for the production of an unshrinkable finish), silk, Celanese, and viscose fabrics dried over phosphorus pentoxide for 5 days contained 0·8%, 1·0%, 2·0%, 1·1%, 0·1%, and 2·2% of moisture and these regained moisture at similar rates when exposed to a moist atmosphere, there being a tendency for the moisture content to rise with increasing rapidity as the humidity rises so that their moisture contents at 100% relative humidity become very large and indefinite in amount. At a normal humidity of 70%, typical samples of fabric contained 6% to 12·4% of moisture in the order Celanese, cotton, linen, silk, wool, viscose. The regain of moisture by viscose is similar to that of wool, and chlorination of wool does not appreciably affect its regain. The initial rates of regain of moisture by dry fabrics are very rapid. Fabrics dried over phosphorus pentoxide showed a high rate of absorption of moisture when exposed to an atmosphere of 50% relative humidity, equilibrium being obtained within 1 hr. (cotton), 3 hrs. (Celanese), and 5—10 hrs. (wool, silk, and viscose silk). Cotton and linen showed a slight increase in bursting strength with increasing humidity, the increase for linen being over 25% when wet. Under similar conditions all the other fabrics behaved similarly to one another in sustaining a large and fairly regular decrease in strength with increase in humidity. Wool, silk, and Celanese fabrics are more weakened by boiling for 1 hr. in a 1% solution of sodium oleate than in 1% solution of sodium carbonate. A. J. HALL.

[Comparison of] tensile strengths of textile fibres under wet and dry conditions. J. OBERMILLER and M. GOERTZ (Textilber., 1926, 7, 163—168, 245—246).—The tensile strengths of (air) dry and wet single textile fibres, before and after extraction of their natural fats and waxes by means of benzene, were determined. Average results for 50 to 100 fibres indicate that the ratios of the wet and dry tensile strengths of cotton, wool, natural silk, cuprammonium ("Adlerseide"), viscose, nitrocellulose, and cellulose acetate artificial silks are 110—120%, 80—90%, 75—85%, 50—60%, 45—55%, 30—40%, and 65—70% respectively, and that these ratios and the tensile strengths of individual fibres are not appreciably affected by extraction of the fat. The tensile strengths of wet and dry fibres and also their ratios are considerably decreased when the dry fibres are exposed to a temperature of 75—100° for 87—119 hrs. For example, the ratios of the wet and dry tensile strengths of American Texas cotton, Indian cotton, merino wool, natural silk (degummed), cuprammonium silk ("Adlerseide"), and "Vistra" silk before and after the heat treatment were 114 and 108·1%, 112·4 and 101·7%, 89·4 and 85·5%, 82·3 and 65·8%, 59·7 and 52·0%, 53·4 and 51·7% respectively. A. J. HALL.

Weighting and decreasing the lustre of artificial silk. W. BRUCKHAUS (Kunstseide, 1925, 7, 260—261).—The lustre of artificial silk is decreased so that it more closely resembles that of natural silk by precipitating barium sulphate within the fibres. For example, artificial silk is steeped in a warm 2—3% solution of sulphuric acid and then directly immersed for 20—30 min. in a 3—5% solution of barium chloride at 70°. The resulting silk absorbs dyes evenly, especially in the absence of salt, and the barium sulphate does not dust out. Artificial silk may be satisfactorily weighted 3—5% by means of barium sulphate but excellent weighting up to about

185% may be obtained by means of the ordinary methods employed for natural silk using stannic chloride, sodium phosphate, and sodium silicate; the resulting silk absorbs dyestuffs evenly.

A. J. Hall.

Spruce wood. H. Urban (Cellulosechem., 1926, 7, 73—78).—Willstätter and Zechmeister's method of separating lignin from wood is modified by using a mixture of 3 vols. of ordinary concentrated hydrochloric acid (*d* 1·18) and 1 vol. of phosphoric acid (*d* 1·7), the wood being treated for about 2 days at 20°. The mixture dissolves cotton wool in 1½ min., and it has the advantages that the lignin is obtained almost free from chlorine, filters readily, and that the inconvenience of handling super-concentrated hydrochloric acid is avoided. Spruce wood, extracted with a mixture of alcohol and benzene, and containing 4·7% OMe, yielded 20—26% of lignin of the composition C 64·0, H 6·1, OMe 15·45, Cl 0·33%, which is expressed by the formula $C_{39}H_{36}O_{12}(OMe)_4$. This is best methylated by suspending 3 g. in 100 c.c. of 45% potassium hydroxide and adding 100 c.c. of methyl sulphate during 10 hrs. with cooling. After one such treatment the MeO-content is 31·3%, and after two treatments the maximum of 32·4% is reached. This corresponds with the introduction of five methoxyl groups, making the formula of the original lignin $C_{39}H_{29}O_6(OH)_5(OMe)_4$, it being assumed that 1 mol. of water is lost. Fusion with potassium hydroxide yields 14% of protocatechuic acid. Cellulose is readily methylated by the above procedure. 2 g. of raw cotton, previously treated with acetone and water, after two methylations with 200 c.c. of 45% potassium hydroxide and 200 c.c. of methyl sulphate, contains 44·7% of OMe, which remains unchanged on further methylation. Methylated cotton retains its structure; it dissolves in chloroform and tetrachloroethane to clear, viscous solutions, but is insoluble in water, alcohol, ether, ethyl acetate, acetone, carbon tetrachloride, and carbon disulphide. Sulphite-cellulose requires nine methylations to reach its maximum of 41·2% OMe. Wood reaches its maximum of 41·0% OMe in five methylations. This, and also methylated wood containing only 20% OMe, yield, on hydrolysis with hydrochloric and phosphoric acids, a methyl-lignin containing 25% OMe. By methylating wood once, hydrolysing, and remethylating the residue a 20% yield of methyl–lignin containing 32·0% OMe is obtained. Although in fully methylated wood the carbohydrate part must be highly methylated, only a small proportion of it can be extracted with chloroform, and the proportion of lignin in the residue is not greatly increased. This suggests combination between the carbohydrate and the lignin. Some of the extracts show a tendency to crystallise. By treating lignin with sodium hydroxide and *p*-toluenesulphochloride two hydroxyl groups (per C_{39}) are readily attacked and a third after repeated treatment; this agrees with the ready methylation of lignin to 24% OMe corresponding with an additional 2½ OMe groups. Methyl-lignin is not affected by *p*-toluenesulphochloride. Wood can be completely dissolved by heating at 75° in acetic anhydride and zinc chloride and passing chlorine through the mixture; the product is precipitated by pouring into water. After such treatment sulphite-cellulose contains 3% Cl and 44·3% of acetyl, and lignin contains 31·6% Cl and 16·2% of acetyl. Wood then contains 11·0% Cl and 34·4% of acetyl, which agrees with the composition calculated on the assumption that it consists of 70% of cellulose and 30% of lignin.

A. Geake.

Wetting spruce wood with calcium and magnesium bisulphite liquors. C. G. Schwalbe and K. Berndt (Papier-Fabr., 1926, 24, 250—253).—When wood shavings (44×2×7 mm.) are shaken with bisulphite solution, containing 3·60% SO_2 and 0·959% MgO or 1·070% CaO, at 70°, the weight absorbed is 126—137% after 3 hrs. and 140—150% after 8 hrs. There is no significant difference between magnesium and calcium bisulphites. The unabsorbed solution is somewhat poorer in sulphurous acid and in base than the original liquor, even when allowance is made for loss of sulphur dioxide into the space above the liquid (which was filled with carbon dioxide to avoid oxidation) and for dilution by the moisture contained in the wood.

A. Geake.

Indicators for the cellulose industry. E. Öman (Papier-Fabr., 1926, 24, 267—270, 285—288, 299—303).—Free sulphurous acid in sulphite liquors may be determined by titration with 0·1*N*-sodium hydroxide to the stage of hydrogen sulphite or of sulphite. For the former titration the best indicator is bromophenol-blue, which has a marked and sharp colour change at the required p_H (4·2). This titration is comparatively little affected by the other weak acids present, viz., carbonic, acetic, and formic acids, and the amounts of calcium sulphite calculated from the titre are accurate to within 1%. For titration to complete neutralisation thymolphthalein is a better indicator than phenolphthalein. With the latter the colour change takes place at p_H 8·2, instead of 9·3, which corresponds with exact neutralisation; the end point also is not very sharp and depends on the amounts of water and of indicator added. White liquor from sulphate-cellulose manufacture may contain sodium hydroxide, sulphide, carbonate, sulphate, and sulphite, and may be titrated with acid in three stages. At p_H 11 the sodium hydroxide and half the sodium sulphide are neutralised, at p_H 9 half the sodium carbonate is also neutralised, and at p_H 4 the sodium sulphide and carbonate are completely neutralised and also half the sulphite. The best indicators for the three stages are Nile-blue, thymol-blue and bromphenol-blue respectively. Frequently the determination of sodium hydrosulphide by titration with iodine, and of sodium hydroxide by titration with hydrochloric acid in the presence of Nile-blue, is sufficient. Mixtures of sodium hydrosulphide and sulphite may be analysed by titrating with iodine, followed by titration of the acid thus liberated. The acid liberated is equivalent to the iodine consumed in oxidising sodium sulphite, and to half that consumed in oxidising sodium hydrosulphide. Two successive titrations of the same sample using different indicators are not recommended;

it is preferable to take separate samples for each titration. Titration of the supernatant liquid over a precipitate such as barium carbonate is also liable to yield inaccurate results. A. GEAKE.

Ripening of viscose solutions. J. D'ANS and A. JÄGER (Kunstseide, 1926, 8, 17—19, 43—46, 57—59 ; Chem. Zentr., 1926, I., 3108).—The number of thiocarbonate groups combined with cellulose may be determined by titration with iodine. The xanthate content diminishes regularly, the velocity increasing with rise of temperature. All carbon disulphide combined with cellulose reacts with iodine. The sensitiveness of a viscose solution to electrolytes depends on the molecular complexity of the cellulose, the number of xanthate groups combined with cellulose, the amount of electrolytes which are always present in viscose solutions, the amount of free sodium hydroxide, and the temperature. In the determination of ammonium chloride ripeness the sodium hydroxide content is important, and the appearance of spontaneous coagulation is not sharply defined. Loss of carbon disulphide molecules does not necessarily imply a diminution in the size of the molecule. In following changes of viscosity the sodium hydroxide content must be the same as that of technical viscose solutions. Viscosity and ammonium chloride ripeness give comparable figures for the condition of viscose solutions. Fresh viscose solution, after mixing with ammonium chloride, generally contains free caustic alkali, the amount of which increases the older the solution. At the coagulation point perfectly fresh viscose solution, containing little alkali, has an excess not of free alkali, but of ammonium chloride. With increasing alkali content of the viscose solution the amount of ammonium chloride used in double decomposition is so great that the cellulose complex is precipitated in a solution which still contains alkali. The stability of viscose solutions towards coagulation increases with increasing xanthate content, and this effect is greater than the influence of sodium hydroxide, especially in fresh solutions. Solutions rich in sodium hydroxide, and containing the same amount of xanthate, have an apparently smaller ammonium chloride ripeness. In very ripe solutions, which almost coagulate spontaneously, the effect of the xanthate content on the ammonium chloride ripeness almost disappears. The number of xanthate groups found by titration with iodine increases with the amount of carbon disulphide used. A. GEAKE.

Increasing the suspension of [paper] coating mixture by the addition of colloids. B. K. STEADMAN (Pulp and Paper Mag., 1926, 24, 307).—Attempts to preserve the suspension of casein coating mixtures by adding other substances of a colloidal nature showed that sodium silicate is effective and fairly satisfactory, but tends to increase the viscosity of the mixture to an undesirable extent. Gum arabic has a similar action, but causes pin-holes to appear in the coating. D. J. NORMAN.

PATENTS.

Washing or otherwise treating wool and other fibrous textile materials. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (E.P. 230,808, 18.2.25. Conv., 11.3.24).—Wool, silk, and other textile materials are passed successively through a series of washing units, each unit being followed by a squeezer. The detergent liquor squeezed from the material leaving each unit flows, with or without partial purification by centrifugal separation of fatty substances and solids, to the adjacent unit containing dirtier liquor. The washing units are very small (about 5 litres capacity) so that they merely ensure the thorough impregnation of the textile material with washing liquor. The period of contact of the washing liquor with the textile materials is thus small, and the detergent power of the liquor is thereby increased. A. J. HALL.

Manufacture of felt. C. and E. PICHARD (E.P. 248,343, 4.2.26. Conv., 28.2.25).—Carroting is effected by treating the hair before or after removal from the skin with a solution of carbamide or derivatives thereof, preferably the nitrate. D. J. NORMAN.

Fabrics or materials and the manufacture thereof. BRIT. CELANESE, LTD., T. C. WOODMAN, and W. A. DICKIE (E.P. 249,946, 10.1.25).—Waterproof and gasproof properties and other special effects may be imparted to fabrics composed of filaments or fibres of esters or ethers of cellulose, either alone or in association with other fibres—animal, vegetable, or artificial—by subjecting the fabric to heat and pressure in order to cause a partial or complete coalescence of the fibres. Plasticising agents such as monomethylxylenesulphonamide facilitate the process, and may either be added to the spinning solution or dissolved in a volatile solvent, which is a non-solvent of cellulose derivatives, *e.g.*, benzol, and sprayed or otherwise distributed over the fabric before the heating operation ; 20 pts. of monomethylxylenesulphonamide dissolved in 100 pts. of benzol is a suitable quantity per 100 pts. of cellulose acetate. The temperature, pressure, and duration of the treatment depend on the extent to which it is desired to close up the pores of the fabric ; for example, the fabric may be passed slowly between calender rolls at 100—180° under pressures varying from 300 to 600 lb. per sq. in. D. J. NORMAN.

Manufacture of artificial materials [from *N*-substituted cellulose thiourethanes]. L. LILIENFELD (E.P. 248,994, 9.7.25. Conv., 30.5.25. Addn. to 231,806 ; B., 1925, 985).—Artificial filaments and films of high lustre and of good tensile strength, both in the wet and dry state, are obtained from *N*-substituted cellulose thiourethanes by using strong acids, *e.g.*, sulphuric acid of 25—65% strength, phosphoric acid of 45—70% strength, hydrochloric acid of 20—35% strength, or acetic acid of 70—100% strength, as precipitating agents instead of the weak acids and other coagulants suggested in the original patent. Neutral or acid salts or organic compounds such as glucose or glycerin may be added to the acid bath, the temperature of which may be below, at, or somewhat above (*e.g.*, 30°) the ordinary temperature. The freshly-precipitated threads are plastic and may be stretched before they are fixed by washing out the

acid. Increased tensile strength is imparted by steaming the washed threads either before or after drying. A suitable spinning solution contains 10—16% of an *N*-substituted cellulose thiourethane (prepared as described in E.P. 231,801; B., 1925, 955) in 6—8% caustic soda solution. The spinning bath may then consist of 50% sulphuric acid maintained at the ordinary temperature or at 5—8°.

D. J. Norman.

Spinning viscose. J. C. Hartogs (U.S.P. 1,573,062, 16.2.16. Appl., 30.6.24).—Evolution of hydrogen sulphide from the viscose spinning bath is avoided by adding a ferric salt, which is decomposed to sulphur and ferrous sulphate. The presence of sulphur in the thread prevents it from breaking in the twisting process, while ferrous sulphate increases the lustre of the threads and prevents their adhesion.

R. B. Clarke.

Cellulose nitrate composition. S. J. Carroll, Assr. to Eastman Kodak Co. (U.S.P. 1,580,189, 13.4.26. Appl., 9.7.24).—A film-forming composition, substantially free from camphor and from high-boiling esters used as substitutes for this, consists of nitrocellulose, monochloronaphthalene, and butyl alcohol. A. Geake.

Artificial silk from viscose. W. Mendel, Assr. to S. A. Neidich (U.S.P. 1,580,843–4, 13.4.26. Appl., 28.5.25).—Viscose is projected (A) into a soft coagulating medium and then into an aqueous phosphate solution, or (B) directly into an aqueous phosphate solution. A. Geake.

Solvents for nitrocellulose and cellulose acetate. Pathé Cinéma (Anc. Etabl. Pathé Frères) (F.P. 601,546–7, 30.10.24).—Mixtures of an alcohol with acetone or with the acetic or formic ester of the alcohol are used as solvents for (A) nitrocellulose, or (B) cellulose acetate, for the manufacture of threads, films, varnishes, or artificial leather. In examples nitrocellulose is dissolved in a mixture of 80 pts. by vol. of absolute alcohol and 20 pts. of acetone, or of 80 pts. of amyl alcohol with 20 pts. of amyl acetate. A. Geake.

Manufacture of lignone derivatives and their application. C. F. Cross and A. Engelstad (E.P. 248,834, 10.12.24).—New lignone derivatives are obtained by the action of acid condensing agents, *e.g.*, hydrochloric acid, either alone or in the presence of phenolic compounds, on lignone or its derivatives, more particularly those sulphonated derivatives resulting from the acid digestion of lignocellulose by the processes described in E.P. 202,016 and 229,002 (B., 1923, 971 A; 1925, 312). The new lignone derivatives may be used in the dyeing industry, or as a basis for the manufacture of writing and printing inks. Example: 100 pts. of lignone extract containing about 37 pts. of solid matter are mixed with 10 pts. of hæmatein and 10 pts. of concentrated hydrochloric acid and heated at 95—100° until the mixture occupies about one-half of its original bulk. The resulting product may be used in the dyeing industry in the same manner as ordinary logwood extract. Writing ink is made, for example, as follows:—A mixture containing 100 pts. of extract (37% of solid matter), 5 pts. of pyrogallol, 2·5 pts. of gallic acid, and 6 pts. of concentrated hydrochloric acid is evaporated at 95—100° to one-half of its original bulk; an equal volume of water is added and in this solution are dissolved successively 10 pts. of crystalline ferrous sulphate and 2 pts. of a soluble blue, such as Nigrosine. The resulting concentrated ink is diluted 7—9 times with water when required for use as a writing ink. The fluidity of the ink may, if desired, be regulated by the addition of a gum. Inks made in this manner tend to become fixed in an insoluble state on drying; if, however, this property is not required, the concentrated lignone extract may be used without previous treatment with condensing agents. D. J. Norman.

Digestion of paper pulp or the like and concentration of the spent lye. J. Holmes (E.P. 248,926, 13.3.25).—The installation comprises a digester, a closed feed reservoir provided with connexions to the top and bottom of the digester, and a multiple-effect evaporator for concentrating the liquor from the reservoir. The first stage of the evaporator is heated by the steam from the digester and reservoir, succeeding stages by the steam from the preceding stage. Heat exchangers placed in the steam connexions between the stages of the evaporator supply heat to the wash liquor and to fresh caustic liquor. If desired the reservoir may be externally heated. Provision is made for introducing steam direct into the first stage of the evaporator. D. J. Norman.

Gas and heat recovery from [wood pulp] digester blow-off exhaust vapours. L. B. Decker (U.S.P. 1,576,643, 16.3.26. Appl., 22.6.25).—The exhaust vapour from the digester is passed into a chip bin which is arranged to communicate with the digester inlet. D. J. Norman.

[Sulphite-pulp] digester. J. W. Alstyne (U.S.P. 1,579,261, 6.4.26. Appl., 3.10.25).—A digester for making sulphite wood pulp has a conical base fitted at the lower end with a discharge pipe; a steam inlet pipe passes centrally up this discharge vent and extends into the bottom of the digester.

D. J. Norman.

Hydrating cellulose fibres [for paper making]. J. A. de Cew, Assr. to Process Engineers, Inc. (U.S.P. 1,580,814, 13.4.26. Appl., 7.6.24).—Fibres are beaten with a solution of an alkali until pliable, and the alkali is then neutralised with aluminium sulphate. A. Geake.

Production of pulp and other products from wood. L. Bradley and E. P. McKeefe (U.S.P. 1,581,671, 20.4.26. Appl., 3.11.21).—Wood is treated with a hot aqueous acid to convert a part of the water-insoluble constituents into fermentable sugars, without cooking the wood or injuring its pulping properties. After separating the sugars, the wood is subjected to a chemical pulping operation.

A. Geake.

Producing paper pulp. E. Lambert and W. E. Matthews (U.S.P. 1,582,336, 27.4.26. Appl., 26.8.22).—Wood stock is comminuted, boiled for 1 hr. with sufficient water to cover it and 1 lb. of potassium hydroxide per 100 lb. of stock, washed to remove dirt, and re-ground. A. Geake.

Stencil sheets. S. Horii (E.P. 250,798, 10.7.25). —A coating for a stencil sheet of fibrous material consists of a solution of an ester or mixture of esters of polysaccharides, such as starch acetates or cellulose acetates, nitrates, or xanthate in suitable solvents such as alcohol, ether, or amyl acetate, to which naphthenic acid glycerides and other tempering agents such as fatty acids, fats, paraffin, ceresin, or other waxes are added. Such a coating is soft but not sticky, stores well, and does not become hard in cold weather. A. Geake.

De-inking paper stock. J. E. Plumstead, Assr. to Jessup & Moore Paper Co. (U.S.P. 1,576,994, 16.3.26. Appl., 16.11.25).—The disintegrated stock is agitated with an alkali; chlorine is then introduced and the resulting mixture subjected to heat. D. J. Norman.

Waterproofing cellulose, paper, and the like. R. Wolffenstein and A. Marcuse (G.P. 426,428, 30.5.14).—Cellulose and materials containing it are waterproofed by treatment with thionyl chloride. A. R. Powell.

Economical disposal of waste sulphite [cellulose] liquor. A. M. Thomsen, Assr. to Crown Willamette Paper Co. (U.S.P. 1,582,317, 27.4.26. Appl., 13.5.24).—Waste liquor from sulphite-cellulose manufacture is concentrated to a syrup and treated with a porous, combustible material. Impregnation of the material with the syrupy liquid is assisted by the former being hotter than the latter. A. Geake.

Manufacture of a non-deliquescent body from sulphite-cellulose [waste] liquor. A. G. Bloxam. From A.-G. f. Anilin-Fabr. (E.P. 251,019, 22.1.25). —See F.P. 592,119; B., 1926, 188.

Making pulp for paper. B. S. Summers (U.S.P. 1,584,902, 18.5.26. Appl., 22.12.23).—See Can. P. 246,537; B., 1926, 153.

De-inking printed paper. L. E. Granton (U.S.P. 1,585,092, 18.5.26. Appl., 26.3.24).—See E.P. 222,160; B., 1924, 977.

Apparatus for drying fabrics, paper, etc. J. J. Lyth (E.P 251,470, 29.6.25).

Preparing rubber-coated fabrics and apparatus therefor. Dunlop Rubber Co., Ltd., and W. J. Dexter (E.P. 251,357, 2.2.25).

Utilising waste heat from gas from sulphur burners etc. (U.S.P. 1,581,511).—See VII.

Combinations of rubber and paper (U.S.P. 1,567,646).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Conditions governing the bleaching of wool with hydrogen peroxide. S. R. and E. R. Trotman (J. Soc. Dyers and Col., 1926, 42, 154—157).—A determination of the factors which are responsible for the loss of weight and damage to the epithelial scales of ordinary and moderately chlorinated wool fabric during bleaching at 55° by means of solutions of sodium peroxide having a known p_H of 9 to 13. The bleaching solutions were prepared by the addition of sodium silicate to hydrogen peroxide. Bleaching causes no marked increase in the proportion of damaged fibres of ordinary wool but a distinct increase in chlorinated wool, especially when the bleaching liquor has a p_H exceeding 10, the increase being proportional to the p_H. The loss of weight produced by bleaching increases with increase of p_H and is much greater for chlorinated than for ordinary wool. A better white is obtained in the more alkaline bleaching liquors and the best colour, together with the least structural damage to the fibres, is obtained with p_H 10. A mixed indicator containing dinitrodiazoaminobenzene (cf. Brandt, B., 1901, 711) and phenolphthalein or phenolthymolphthalein is suitable for controlling the acidity of bleaching liquors. For example, an indicator containing 5 c.c. of a saturated alcoholic solution of dinitrodiazoaminobenzene and 0·1 c.c. of a 0·4% alcoholic solution of phenolphthalein gives a range of colours: p_H 8 yellow, p_H 9 reddish-yellow, p_H 9·5 redder yellow, p_H 10 orange, p_H 11 red, and p_H 12 deeper red. A. J. Hall.

From "hot-bleach" to "cold-bleach." [Development of the Mohr process of bleaching.] R. Mohr (Textilber., 1925, 6, 909—912).—The Mohr process of bleaching in which cellulosic textile materials (fabric may be treated in open width) are successively bleached under a hydraulic pressure of 2—3 atm. in the same container with a chlorine bleaching solution, soured, then further bleached with a liquor containing a peroxide and afterwards thoroughly washed, is more economical in labour and particularly lime than the usual processes of kiering and bleaching with hypochlorites. Cold or warm liquors may be used; the textile material suffers only a small loss in weight and is uniformly and thoroughly purified so that it is very absorbent and takes up dyes evenly. A. J. Hall.

[Comparison of the] "hot-bleach" and "Mohr-bleach" processes. M. Freiberger (Textilber., 1926, 7, 148, 226—227).—The advantages claimed for the Mohr process of bleaching (cf. preceding abstract) are refuted. The Mohr process removes pectic and protein impurities from cotton, but only partially removes fats and waxes. The last-named impurities retard penetration of the cotton by the bleaching liquors so that the use of excess of bleaching agents is necessary; a considerable amount of oxycellulose is thereby formed. Cotton bleached by the Mohr process becomes yellower after extraction with light petroleum and the extracted cotton becomes still yellower when

steamed. The yellowing of the bleached cotton during storage is hindered by the presence of the fats and waxes incompletely removed during bleaching. Cotton fabric suffers a 5% loss of weight during bleaching by the Mohr process whereas the "hot-bleach" process of Thies–Herzig produces a loss of 12%; fabric bleached by the Mohr process thus contains impurities, which are revealed by dyeing it with Alizarin. The Mohr process of bleaching produces an excessive loss of strength and is really but a partial bleach which can be better obtained by the usual methods or in less elaborate plant.

A. J. Hall.

Test for mercerised cotton. H. Mennell (J. Text. Inst., 1926, **17**, T 247).—The test depends on the fact that mercerised cotton is rendered sensitive to substantive dyes by treatment with sulphuric acid, especially in the presence of formaldehyde. The reagent consists of 320 c.c. of sulphuric acid of *d* 1·6, diluted with 260 c.c. of 40% formaldehyde, and the dye preferred is Chlorazol Sky Blue G.W., used in a boiling bath made alkaline with sodium carbonate. The sample to be tested, and samples known to be mercerised and unmercerised respectively, are steeped in the acid for 2 min., then washed and dyed. If the dye-bath is of such a concentration as to give a 0·1% shade on unmercerised cotton it will give about a 0·8% shade on fully mercerised material, and the degree of mercerisation can be judged from the intermediate shades. Dyed material may be stripped with hypochlorite or hyposulphite before testing.

J. C. Withers.

Simple and reliable test for mercerisation. R. W. Kinkead (J. Text. Inst., 1926, **17**, T 213—219).—The material is stained with Methylene Blue and then boiled with sodium carbonate and a trace of iodine. Details of concentrations, which are varied slightly for different cellulosic fibres, must be observed. If the material has not been mercerised it will remain blue, but if mercerised with caustic alkalis it will become reddish-purple, and the new shade will afford a clue to the experienced observer of the concentration of the alkali employed. The test may be applied to a dyed material after stripping and is not affected if the fibre has been mildly bleached or soaked in acids. If the mercerisation has been carried out by means of acids, however, the colour change will be much less definite, but such materials respond readily to Huebner's iodine tests (J.S.C.I., 1908, **27**, 105).

J. C. Withers.

Dyeing of wool with indigo. F. Peterhauser (J. Soc. Dyers and Col., 1926, **42**, 152—154).—Indigosol O (cf. Vaucher and Bader, B., 1924, 864) is superior to indigo since it is easily soluble in water and may be applied to wool by the methods used for acid dyes; it does not oxidise in the dye-bath and allows the accurate matching of desired shades. Its subsequent development by oxidation is not satisfactory when hydrogen peroxide, a persulphate, or atmospheric oxygen is used. Indigosol OR and O4B are similar soluble derivatives of monobromo- and tetrabromo-indigo respectively.

A. J. Hall.

Influence [on dyeing] of the degree of dispersion of dyes in dye liquors. R. Roestel (Textilber., 1926, **7**, 228—230).—More uniform and deeper shades are obtained on vegetable, Chardonnet silk, and wool fibres by means of direct, mordant, basic, vat, and sulphur dyes when a dispersing or wetting-out agent, particularly Tetracarnite (Sandoz), is added to the dye liquors. Chardonnet silk is satisfactorily and evenly dyed with the insoluble bases of basic dyes such as Chrysoidine G extra, Rhoduline Heliotrope, and Methylene Blue RR dispersed in aqueous solutions containing Tetracarnite, and under similar conditions the shade obtained on wool by means of the base of Brilliant Green A extra is brighter than, and as fast to rubbing as, that obtained by dyeing wool in the usual manner in the presence of acetic acid. The presence of Tetracarnite assists the dyeing of cotton with Alizarin more than similar additions of ammonia or Turkey-red oil. Benzo Fast Red L dyed on cotton is not appreciably affected by treatment with boiling aqueous solutions containing 10% of sodium carbonate or 33% of Tetracarnite or 10% of Turkey-red oil and 10% of sodium carbonate, but under similar conditions dyeings of Columbia Blue 3G are considerably stripped. Benzo Fast Red L and Patent Blue are readily removed from wool by treatment with a 50% solution of Tetracarnite but are much less affected by similar treatment with aqueous solutions containing sodium carbonate or Glauber's salt and sulphuric acid. A. J. Hall.

Dyeing materials used for the manufacture of buttons and the like. Flemming (Deuts. Färber-Ztg., 1925, [2]; Textilber., 1926, **7**, 175—176).—For the production of satisfactory shades on horn, the material is steeped overnight in a 10% solution of sodium carbonate, then washed in water, mordanted by immersion for 6—8 hrs. in a solution containing one or more of the salts of copper, iron, and chromium, and afterwards dyed in a cold solution containing a suitable oxidation fur dye (*e.g.*, *p*-phenylenediamine and hydrogen peroxide); mordanting may be effected before or after dyeing and prevents subsequent sublimation of dye from the horn. Ivory is dyed by methods similar to those used for horn but ivory nut materials may be dyed in boiling liquors containing basic, direct, or sulphur dyes. Ivory is also dyed with oxidation colours by the method described above. Galalith (prepared from casein) is preferably dyed in warm solutions containing acid or direct dyes, whereas Bakelite materials (phenol-formaldehyde condensation products) are satisfactorily coloured by means of basic dyes. A. J. Hall.

Chemical analysis of cotton. Absorption of Methylene Blue from buffered solutions. Clibbens and Geake.—See V.

Chemical analysis of cotton. Scouring losses. Fargher and Higginbotham.—See V.

PATENTS.

Raw-stock-dyeing apparatus. F. M. and G. W. MORTON (U.S.P. 1,571,863, 2.2.26. Appl., 6.4.25).—The dye-vat has a dye-liquor inlet port in the upper portion of one side wall and a removable curved cover plate to which a perforated dye-liquor distribution plate is attached on its under side. The lower side of the inlet port is inclined inwards and upwards, the upper side being horizontal and the other sides diverging outwards. The edge of the dye-liquor distribution plate is inclined downwards so that it is in line with the upwardly inclined lower side of the inlet port. Dye-liquor entering through the inlet port is deflected upwards by the edge of the distribution plate and afterwards percolates downwards through the plate on to textile material below. A. J. HALL.

Dye vat. F. M. and G. W. MORTON (U.S.P. 1,571,864, 2.2.26. Appl., 26.10.25).—The vertical side walls of the vat are attached by means of an outer flange to a horizontal bottom plate provided with a horizontal dye-liquor outlet port. A horizontal perforated plate in the same plane as the outer flange rests on lugs projecting upwards from the bottom plate, and the latter is inclined downwards from the centre to its outer edges. A. J. HALL.

Producing fast dyeings on wool. BADISCHE ANILIN- & SODA-FABR., Assees. of H. KRZIKALLA, H. KÄMMERER, and J. NÜSSLEIN (U.S.P. 1,579,121, 30.3.26. Appl., 3.8.25).—Wool or other animal fibre is impregnated with a sulphonated azo-dye component containing an amino- or hydroxy-group or both so that it can be coupled on the fibre with a diazo-compound. To obtain a fast colour the resulting dye must be difficultly soluble, hence the number of sulphonic groups must be reduced to a minimum. Chromed and unchromed dyes which will couple may also be employed to impregnate wool. For example, wool is boiled for 1 hr. in a bath containing 1:5-di-[6-sulpho-2-hydroxy-3-naphthoylamido]naphthalene, and is then treated with diazotised *m*-xylidine when a bluish-red shade fast to washing, milling, and light is developed. T. S. WHEELER.

Dyeing brown shades on [cotton] fibres. COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIS, ETABL. KUHLMANN (F.P. 595,705, 27.6.24).—Cotton is dyed in brown shades fast to washing, alkali, chlorine, and light by impregnation with a solution containing products obtained by the condensation of formaldehyde and aniline or its homologues in acid solution, being afterwards passed through a solution of caustic soda or sodium carbonate, then washed, oxidised with a dilute solution of sulphuric acid containing a dichromate or persulphate, washed, and soaped. The resulting shades may also be diazotised and developed with β-naphthol or coupled with diazo-compounds. Alternatively cotton is impregnated with a solution containing lactic acid, the above described condensation products, sodium chlorate, sodium acetate, and copper sulphate or a vanadium salt, then oxidised during 6 hrs. at 40° with a solution of a dichromate; white and coloured resists may be obtained by after-printing with a reducing agent, *e.g.*, a sulphite, bisulphite, or hyposulphite, and suitable dyes. Brown effects are also obtained by printing cotton fabric with a paste containing the condensation products and gum tragacanth thickening, drying, fixing in an alkaline solution, and developing the shade by oxidation with a persulphate; alizarin and vat dyes may be printed and developed simultaneously by oxidation. A. J. HALL.

Dyes and dyeing process. BADISCHE ANILIN- & SODA-FABR. (F.P. 599,566, 8.5.25. Conv., 30.6 and 24.7.24 and 21.1.25).—Fast dyeings on cotton and artificial silks (cellulose acetate silk excepted) capable of being after-treated with zinc chloride, lead acetate, etc., are obtained by direct application in the presence of sodium chloride or Glauber's salt of suitable substantive or non-substantive dyes which have been previously treated with chromium salts, or by the application of suitable dyes in the presence of chromium salts (*e.g.*, fluoride, acetate, oxalate, and formate). Suitable dyes include the chromium compounds of azo dyes prepared from mixtures of 2:5- and 2:8-naphthylaminesulphonic acids and salicylic acid (yellow); from *m*-aminobenzoyl-*p*-aminosulphosalicylic acid and 1:8-acetaminonaphthol-4:6-disulphonic acid (red); and from benzidine, *oo'*-dianisidine, or diaminodiphenylcarbamide and salicylic acid (golden-yellow to yellowish-orange). The chromium compound of the triphenylmethane dye, Chromoxan Blue R, dyes blue. A. J. HALL.

Azo dyes [for cellulose acetate]. BADISCHE ANILIN- & SODA-FABR. (F.P. 600,106, 26.6.25. Conv., 4.9.24).—Cellulose acetate silk is dyed in fast shades from aqueous solutions or suspensions of azo dyes containing one or more monohydroxyethylamino-groups, but no sulphonic acid group. A suitable dye is obtained by coupling diazotised *p*-nitroaniline or 3-nitro-*p*-toluidine with hydroxyethylaniline in acetic acid solution. A. J. HALL.

Formation of pigment azo dyes on vegetable fibres. L. CASSELLA & CO., Assees. of G. KALISCHER and K. KELLER (G.P. 422,467, 12.4.24).—Very deep shades fast to rubbing are obtained by impregnating cotton with azoxy-derivatives (related to azo-derivatives) of arylides of β-hydroxynaphthoic acid in alkaline solution and afterwards coupling with a diazo-compound. A suitable azoxy-compound is obtained by alkaline reduction of the nitroanilide of β-hydroxynaphthoic acid. A. J. HALL.

Dyeing artificial silks manufactured from acidyl celluloses, cellulose ethers, and related products. FARBENFABR. VORM. F. BAYER & CO., Assees. of W. DUISBERG, W. HENTRICH, and L. ZEH (G.P. 423,601, 25.12.23. Addn. to 418,940; cf. E.P. 225,862; B., 1925, 583).—Azo dyestuffs suitable for dyeing cellulose acetate silk are prepared by coupling diazotised amines or their substituted

derivatives with aralkylarylamines or their substituted derivatives containing carboxylic or sulphonic acid groups. For example, yellow, bordeaux, and orange-red dyes are obtained by coupling 5-nitro-2-anisidine and benzyl-*o*-sulphanilic acid, 2:4-dinitroaniline and 4-sulphobenzyl-2-toluidine, and 2:4-dinitroaniline and benzylanthranilic acid respectively.
A. J. Hall.

Manufacture of effect threads. Farbenfabr. vorm. F. Bayer & Co., Assees. of G. Rudolph (G.P. 423,602, 27.3.24. Addn. to 407,834; B., 1925, 240).—Fabrics capable of being dyed in more than one colour are woven from ordinary silk and from silk yarn having a diminished affinity for dyestuffs produced by treatment for 2 hrs. with tannic acid and then for 1 hr. with tin salts in the presence of formaldehyde. A. J. Hall.

Finishing and ornamentation of textile materials. J. Huebner (E.P. 250,283, 10.10.24).—Cellulose is deposited upon or within textile materials composed wholly or partly of animal fibres, such as wool or silk, by printing, padding, or stencilling such materials with suitable cuprammonium solutions of cotton or other cellulosic substance and afterwards treating them with a precipitating agent, such as hydrochloric acid or caustic soda, the residual copper being removed simultaneously or by a subsequent process; the textile materials are dyed or printed before or after the deposition of cellulose. For example, fabric is printed with a 2% cuprammonium solution of bleached cotton (30 g. of copper per litre of solution), dried (optional), soured in hydrochloric acid of *d* 1·05, whereby the cellulose is deposited and copper removed, and then dyed preferably without previous drying. The effects produced are very close imitations of coloured woven fabrics (cf. E.P. 227,480; B., 1925, 240). A. J. Hall.

Lignone derivatives (E.P. 248,834).—See V.

Starch preparations (E.P. 244,708).—See XVII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Recovery of selenium from lead chamber slimes. W. Stahl (Chem.-Ztg., 1926, 50, 280).—The most economical process for treating the slimes which accumulate in the lead chamber process of making sulphuric acid consists in heating them in cast iron pots with strong sulphuric acid and a little sulphur trioxide, the fumes evolved being returned to the chamber circuit. The reaction product is extracted with dilute sulphuric acid, the insoluble residue separated in a filter-press and smelted for the recovery of lead, and the solution treated with sulphur dioxide for the precipitation of selenium. The acid filtrate from the selenium is concentrated and used again in the process until it becomes too foul. Addition of lime to the foul liquor neutralises the acidity and precipitates calcium sulphate, which may be used for the manufacture of plaster of Paris etc., and the liquor may be safely run to waste.
A. R. Powell.

Sensitiveness of diphenylamine as a reagent for nitric acid. R. Krauer (Chimica e Ind. (Brazil), 1926, **1**, 143—144).—The solution of diphenylamine in sulphuric acid used as a reagent for nitric acid gives, after keeping, a positive reaction with pure sulphuric acid. This is probably due to the absorption of traces of nitric acid from the atmosphere of the laboratory. By using crystals of diphenylamine instead of the solution in sulphuric acid as little as 0·00021% of nitric acid can be detected in sulphuric acid. The reaction is intensified by dilution with water. G. W. Robinson.

Constitution of bleaching powder. H. Ditz and B. Neumann (Z. Elektrochem., 1926, **32**, 231—240).—Polemical between Ditz and Neumann (cf. Neumann and Hauck, B., 1926, 190).
M. S. Burr.

Manufacture of sodium chromate. N. F. Yushkevich (Trans. Inst. Econ. Min. Petr. (Russia), 1925, [13], 1—29; Chem. Abstr., 1926, 20, 1305).—When 37·85% of chrome iron ore (44·6% Cr_2O_3) is roasted with 24·3% of sodium carbonate (98·5% pure) and 37·85% of lime (90% pure), the formation of chromate at 700° is slow, whilst at 1160°, 95% of the chromium is oxidised in 30 min. The heating must be rapid, the stirring continuous, and the temperature of the heating gas must not exceed 1260°. A. A. Eldridge.

Analysis of lime. J. C. Bailar (Ind. Eng. Chem., 1926, **18**, 389—390).—The determination of calcium oxide in the presence of carbonate, *i.e.*, the available lime, may be carried out by adding to a well hydrated portion of the sample, either excess of iodine and titrating with sodium thiosulphate, or excess of zinc chloride and titrating with sodium hydroxide using an indicator consisting of phenolphthalein and alizarin-cyanine-green C. The methods are quicker than the well-known sucrose method, but the iodine method is expensive when many determinations are required. The author considers that the zinc chloride method is the most satisfactory for the sugar industry. G. T. Peard.

Stabilisation of hydrogen peroxide solutions and of perborates. Karsten-Salmony (Chem.-Ztg., 1926, 50, 280—281).—A review of the patent literature. A. R. Powell.

Determination of traces of carbon monoxide. H. Davies and H. Hartley (J.S.C.I., 1926, **45**, 164—168 T).—Iodine pentoxide, prepared by the chloric acid method, is employed and the apparatus and procedure adopted are described. The pentoxide is shown to be stable over the temperature range (120—150°) used in the determination, but, owing to the carrying over of oxy- or hydroxy-compounds of iodine, generally a small positive "blank" is found, even when air free from carbon monoxide is passed over heated iodine pentoxide. The procedure which results in a "blank" not exceeding 1—2 parts per

million is detailed, and the importance of using dehydrated material is emphasised. A special form of absorption vessel for completely absorbing and retaining the iodine liberated during a determination, is described and the method of titration is dealt with.

Determination of small quantities of hydrogen in gaseous mixtures. P. LEBEAU and P. MARMASSE (Compt. rend., 1926, 182, 1086—1087).—The determination of a small percentage of hydrogen in a mixture of gases may be carried out accurately by passing the mixture over silica gel (previously heated at 150° in a vacuum) cooled to the temperature of liquid air. The only gases not fixed by the gel under these conditions are hydrogen, helium, neon, and traces of methane, nitrogen, oxygen, and carbon monoxide. This mixture is then analysed by the method previously described (Lebeau and Damiens, A., 1913, ii, 253, 349: B., 1913, 126, 186, 277). H. J. EVANS.

Influence of rate of stirring on reaction velocity. HUBER and REID.—See I.

PATENTS.

Producing commercial hydrochloric acid. H. A. GALT, Assr. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,581,436, 20.4.26. Appl., 23.3.23).—A concentrated solution of calcium chloride is treated with sulphuric acid of such strength as to produce precipitated calcium sulphate; after leaving the mixture to react, it is filtered to obtain a concentrated solution of hydrochloric acid. H. ROYAL-DAWSON.

Utilising waste heat from gas [from sulphur burners etc.]. H. CLEMM and A. SCHNEIDER, Assrs. to ZELLSTOFF-FABR. WALDHOF (U.S.P. 1,581,511, 20.4.26. Appl., 3.4.22).—The heat of gases from sulphur burners and pyrites furnaces is transmitted to a heat-exchanging device through which fresh sulphite lye is forced under pressure, the lye being then conveyed to an apparatus for boiling cellulose. The whole of the system containing fresh lye, whether heated or not, is completely closed, thus retaining a certain pressure therein in order to avoid any loss of sulphur dioxide and any noxious change of the lye.
H. ROYAL-DAWSON.

Treating waste liquors containing cyanogen compounds. J. DENIS (E.P. 250,824, 18.9.25).—The liquor is treated with lime water and ferrous sulphate, and then with carbon dioxide to precipitate iron ferrocyanide as a blue sludge, which is filtered off. The filtrate is treated with barium hydroxide or the like to precipitate the excess of carbonic acid, the precipitate carrying down the last traces of iron ferrocyanide. When the liquors contain phenols and hydrocarbons these are separated by their difference of density before or after adding the reagents mentioned in the first stage of the process and are conveyed into a closed reaction chamber into which is passed a current of air or other gas, which is carburetted by the phenols and hydrocarbons and may be utilised as fuel gas. H. ROYAL-DAWSON.

Obtaining alkali thiosulphate from solutions containing alkali sulphide. F. RÜSBERG, Assr. to RHENANIA VEREIN CHEM. FABR. A.-G. (U.S.P. 1,567,755, 29.12.25. Appl., 28.5.25).—Solutions containing sodium sulphide are treated with a gas containing 32% by vol. of sulphur dioxide in the presence of sodium sulphite. H. ROYAL-DAWSON.

Manufacturing sodium fluoride from silicofluorine compounds. W. SIEGEL (U.S.P. 1,581,819, 20.4.26. Appl., 9.5.25).—Potassium silicofluoride is treated with an alkaline potassium compound; after separating the silicic acid formed, the potassium fluoride so obtained is further treated with a sodium compound to transform the potassium fluoride into sodium fluoride, which is then separated from the potassium compound. H. ROYAL-DAWSON.

Manufacture of anhydrous chlorides. J. GOHIN (F.P. 601,612, 4.11.24).—Solid chlorides are sublimed in one or two stages without condensation in a chamber provided with a lining resistant to the action of hot chlorine, in such manner that the sublimate commences to condense only at the end of the apparatus. J. S. G. THOMAS.

Separation of barium and radium salts. I. BACHILOV (F.P. 601,938, 7.8.25).—The solution of the double salt of barium and radium is crystallised after addition of salts containing the same ion as the solution, and which do not form salts soluble in the solvent employed, with the barium and radium salts present. J. S. G. THOMAS.

Production of alumina and potassium salts from leucitic rocks. F. GIORDANI and U. POMILIO (G.P. 426,832, 10.9.21).—Solution of constituents other than potassium and aluminium in the decomposition of leucite is reduced to a minimum by treating the rock with a small quantity of acid at first, and adding more acid from time to time as solution proceeds, the total quantity of acid added being somewhat less than that required for complete solution of the aluminium and potassium.
L. A. COLES.

Apparatus for expelling ammonia from ammoniacal liquor. BAMAG-MEGUIN A.-G. (G.P. 426,863, 27.8.24).—The apparatus comprises a series of columns for expelling free ammonia, provided with serrated bells having their overflow devices high up, so that the bells lie completely below the level of the liquid, thereby exposing a large surface for evaporation, and a series of columns for expelling fixed ammonia in which intimate admixture of lime with the liquor is effected by the provision of narrow ducts running parallel to the periphery of the bells. The columns are divided into two sections by a sloping partition, the lower section serving as a preheater and storage chamber for the liquor. Before leaving the columns, the gas passes through a chamber in which liquid particles are separated as completely as possible, *e.g.*, by the provision of baffle plates, the separated liquid passing to the previous column.
L. A. COLES.

Decomposition of material containing selenium. Mansfeld-A.-G. für Bergbau & Hüttenbetrieb, and K. Wagenmann (G.P. 426,669, 22.7.25).—Mixtures of the material with sodium bisulphate, or with sulphuric acid and alkali sulphates, are heated above 700°, and the selenium which distils over is condensed. Anode sludge, before subjection to the above treatment, is dried and heated with sufficient concentrated sulphuric acid to convert into sulphates all the constituents soluble in sulphuric acid.
L. A. Coles.

Production of zirconium oxide. H. S. Cooper and L. P. Bensing, Assrs. to Kemet Laboratories Co. (U.S.P. 1,582,126, 27.4.26. Appl., 1.3.20).—Anhydrous zirconium chloride, free from iron, is hydrolysed and heated at gradually rising temperatures.
H. Royal-Dawson.

Production of barium peroxide. A. F. Meyerhofer (G.P. 426,034, 20.10.23).—Barium phosphate is treated with hydrofluosilicic acid, the precipitated barium silicofluoride separated from the phosphoric acid produced and decomposed by heating into barium fluoride and silicon tetrafluoride, the latter being used for the regeneration of hydrofluosilicic acid whilst the barium fluoride is treated with calcium hydroxide yielding calcium fluoride and barium hydroxide, which latter is then converted into barium peroxide by the usual method.
J. S. G. Thomas.

Manufacture of hydrogen and other gases. Airship Guarantee Co., Ltd., and P. L. Teed (E.P. 250,700, 27.1.25).—In the manufacture of hydrogen from ferrosilicon and caustic soda the residual sludge is continuously discharged into a trap adapted to be raised or tilted to prevent the ingress of air into the generator, while allowing the entire plant to be purged periodically to carry away any air which may leak into the system at other places. Should the pressure in the generator rise above a predetermined limit, the excess gas bubbles through a hydraulic seal into the atmosphere. The gas and steam from the generator pass through a condenser and the condensed water is returned wholly or in part to the generator to keep the strength of the solution more or less constant.
H. Royal-Dawson.

Removing traces of chlorine from air. L. Rosenstein (U.S.P. 1,578,850, 30.3.26. Appl., 19.4.23. Renewed 31.8.25).—Air containing traces of chlorine is passed up a tower containing scrap iron down which a solution of ferrous chloride continuously flows, being withdrawn at the bottom and pumped to the top of the tower. The chlorine rapidly reacts with the iron in presence of ferrous chloride solution forming a further quantity of that substance. At intervals a portion of the circulating solution is removed and fresh iron and water are added.
T. S. Wheeler.

Manufacturing powder of lead suboxide intermingled with powder of metallic lead. G. Shimadzu (U.S.P. 1,584,150, 11.5.26. Appl., 14.7.23). —See E.P. 218,119 ; B., 1924, 711.

Manufacturing lead oxide. G. Shimadzu (U.S.P. 1,584,151, 11.5.26. Appl., 18.4.24).—See E.P. 236,368 ; B., 1925, 714.

Production of lead acetate. R. Plaüeln (U.S.P. 1,585,316, 18.5.26. Appl., 20.5.24).—See E.P. 224,199 ; B., 1925, 283.

Producing substances from boron and carbon. E. Podszus (U.S.P. 1,585,412, 18.5.26. Appl., 15.9.23).—See E.P. 204,337 ; B., 1924, 982.

Ammonia from vinasses (U.S.P. 1,576,427). —See XVII.

VIII.—GLASS; CERAMICS.

Annealing and re-annealing of glass. III., IV. W. M. Hampton (Trans. Opt. Soc., 1925-6, **27**, 161—179 ; cf. B., 1925, 207).—Formulæ previously deduced were applied to the case of heating glass under constant gradient. The rate of heating did not affect the ratio $(S_0\text{-}S)/S$ (S_0=strain due to temperature gradient, S=strain relieved by plastic flow) although it altered S_0-S at a given temperature, whilst changes in the size of the specimen affected both the ratio and S_0-S. The theoretical strain–temperature curve agreed in form with the experimental curve when the coefficient of expansion was taken as variable with temperature. Theoretical analysis indicated that the annealing temperature could only be measured because of the sudden change in the coefficient of expansion in the region where measurable viscous flow is commencing. This temperature was only of use for comparing the annealing requirements of similar glasses, and, because of temperature lag, a standard rate of heating was necessary. The correctness of the stress–temperature equation $df/dt=f^2k/\eta$ was confirmed in tests at relatively high temperatures and the values of k/η deduced for two glasses at various temperatures. From viscosity measurements, from the rate of sag of beams under load, values of k were found in the two cases. At lower temperatures (300°) the disappearance of birefringence was not a phenomenon of viscosity only but of plasticity, in which the law $df/dt=(f-f_L)^2k/\eta$ held. The stress was relieved by flow on annealing until it reached a value f_L, a constant depending on the temperature. This conclusion was in agreement with results given by Adams and Williamson (B., 1921, 81A), it explained Twyman's results on chilling and tempering (B., 1923, 1177A), and it correlated the behaviour of glass with the property of clays and paints as studied by Bingham and Green (B., 1920, 495 A). The viscosity–temperature curve for three soda–potash–lead silica glasses gave a point of inflection as log η increased beyond 13.
A. Cousen.

Progress report on investigation of saggar clays. R. A. Heindl (J. Amer. Ceram. Soc., 1926, **9**, 131—143).—A study of the characteristics of the individual saggar clays used in America is presented. A basis of classification was obtained by determination of the following properties : water of plasticity ; volume and linear shrinkage ; porosity and modulus

of rupture in the dry state ; softening point ; strength, porosity, and shrinkage after firing to five different temperatures ; resistance to dunting ; petrographic and chemical analyses. For the dunting test, small saggars were placed in furnaces at temperatures varying from 350° to 800° for 30 min., then withdrawn quickly and allowed to cool in the air. Most of the saggars fractured at or below 575°. For the purpose of continuing the study, the clays are arranged in five groups in the order of increasing average modulus of rupture and decreasing average porosity. Additional tests to study the effect of repeated firings showed that, after a number of firings under industrial conditions, the general result was an increase in the transverse strength and a decrease in porosity. Petrographic examination, however, indicated that, although the cold modulus of rupture was increased by repeated firings, the increase in the quantity of glass due to this treatment would probably actually weaken the structure at kiln temperatures. F. Salt.

Refractory materials. D. B. Schulz (Feuerfest, 1926, **2**, 33—35).—The paper presents the following data :—(1) Curves showing the relationship between composition and refractoriness of alumina–silica and kaolin–silica mixtures, from results by Seger ; (2) examples of the composition of good average refractory materials (after Koppers) to illustrate the division of these by Littinsky into quartz (acid), chamotte (basic), and quartz–chamotte ; (3) analyses of refractory materials for marine boilers, under the divisions (*a*) highly aluminous, (*b*) "dynaxite," (*c*) normal ; (4) the relationship between the composition and softening and fusion points of refractory materials, after Hirsch ; (5) the analyses of the ash of three naval coals from Wilhelmshaven ; (6) the colour, composition, and m.p. of 11 coal ashes from various localities, by Aktiebolaget Ljungström Angturbin, Stockholm. A. Cousen.

Flow of heat in the walls of ceramic kilns. H. Willmer (Ber. Deuts. Keram. Ges., 1925, **6**, 49—62).—A method of determining directly the loss of heat through kiln walls is presented. Five kilns of different types were studied under working conditions. Thermo-elements were placed in holes, 50 cm. deep, bored into the walls at an average height of 1·8 m. above the kiln floor, and about 10 to 20 cm. apart. The outside temperature of the walls was measured by means of surface thermo-elements—small copper discs, to which thermo-elements were soldered. The weight of the kiln setting (saggars and ware) and the coal consumption were also determined. The results obtained indicated that the amount of heat absorbed by the walls, soles, and crowns of round kilns varied—according to the type and size of the kiln, the firing period, and the maximum temperature attained—from 12·8% to 18·9% of the total heat generated from the coal. The corresponding figure for a gas-fired kiln was 25%. An additional 5% was lost by radiation to the atmosphere from a stoneware kiln. These figures correspond roughly with the amount of heat required to heat the whole of the kiln setting including saggars. The degree of penetration (S) is given by the formula : $S=A\sqrt{z}$, where z is the firing period in hrs., and A a coefficient, which varies for the different kilns from 5·2 to 15·9. To test the effect of insulation, the middle portion of the door of an earthenware biscuit kiln was constructed of bricks made from diatomaceous earth, temperature measurements being taken as before. From the results it is calculated that, with a completely insulated kiln, a saving of 7% in fuel would have been effected. F. Salt.

Absorption of sulphur dioxide from kiln gases by ceramic ware. F. G. Jackson (J. Amer. Ceram. Soc., 1926, **9**, 154—173).—A study was made of two clays, one practically free from sulphur, the other containing pyrites. Draw trials were taken from a laboratory kiln and from industrial kilns at suitable temperature intervals. The laboratory kiln was an exact replica in miniature of a round down-draught kiln and measured 15 in. in diam. 10 g. were removed from the surface of each test brick and analysed for water-soluble sulphates and bases. The results of the tests showed that greatly increased concentration of sulphur in the kiln atmosphere produced only slight increase in the absorption of sulphur by the ware. Hence, variations in the sulphur content of the coal, within industrial limits, can have little effect on the amount of sulphur absorbed by the ware. The greatest absorption took place at about 500°, the time taken to reach this temperature having little effect. Under extreme conditions of exposure to sulphur gases only a small proportion of the bases present in the clays was converted into sulphates. When a coal containing only 0·5% of sulphur was used as fuel, less than 8% of the available sulphur was absorbed by the ware. Decomposition of the sulphates at higher temperatures could not be noticeably affected by the law of mass action, since the concentration of sulphur in the kiln gases is low under all circumstances. F. Salt.

Properties of potters' flints and their effects in white-ware bodies. E. E. Pressler and W. L. Shearer (Tech. Papers U.S. Bur. Standards, 1926, **20**, [310], 289—315).—A study was made of the physical and chemical properties of 15 American and 2 French flints and of the firing behaviour of typical vitreous and semi-vitreous white-ware bodies containing these flints. Grading by air elutriation showed that, with four exceptions, more than 50% of the material consisted of particles less than 0·02 mm. diam., and about 99% of particles less than 0·10 mm. diam. The sp. gr. of the French flints was lower than that of the American and was reduced more by calcination. Calcination to cone 9 caused the development of much cristobalite, cryptocrystalline flints showing a greater proportion than quartz flints. The effect of heat treatment on flints and on pulverised bodies was studied by reference to alundum powder as standard. The resistance of bodies to thermal shock was tested by quenching specimens at 600° in air (expansion due to α–β quartz inversion at about 573°) and at 200° in water at room temperature. The thermal expansion near the quartz inversion point was approximately proportional to the flint content,

other factors remaining constant. Bodies containing cryptocrystalline flint showed greater expansion in the cristobalite inversion range (220—275°) than quartz flint bodies, and less at the quartz inversion point. Extreme fineness of the flint in bodies caused a slightly higher rate of expansion in the cristobalite inversion range and a lower rate at the quartz inversion point. The thermal shock tests indicated that cryptocrystalline flints promote resistance in the temperature range near the quartz inversion point, whereas quartz flints increase the resistance in the cristobalite range. Cryptocrystalline flints do not lower the maturing temperature of a body, but tend to render it more susceptible to overfiring. Finely-ground quartz flint lowers the maturing temperature of a body without affecting the vitrification range.

F. Salt.

Relationship between the constitution and the properties of porcelain. R. Rieke (Ber. Deuts. Keram. Ges., 1925, 6, 144—153).—The structure of a fired porcelain body usually presents the appearance of a glassy (felspathic) matrix, in which crystals of mullite ($3Al_2O_3,2SiO_2$) and quartz are more or less finely distributed, and which is permeated with minute pores due to air bubbles. The mullite and quartz crystals may vary both in number and size. The properties of ordinary hard porcelain are mainly determined by the nature of the matrix, which usually forms much more than one-half of the body. Its composition may vary as follows: 78—86% SiO_2, 11—8% Al_2O_3, 10—6% K_2O, omitting minor ingredients. The composition: 86% SiO_2, 8% Al_2O_3, 6% K_2O represents the optimum theoretical conditions, when all the quartz and 10—15% of the mullite have been dissolved. The nearer a porcelain approaches this optimum in composition, the better its physical properties. The quantity, size, and distribution of the mullite crystals have a marked effect upon the mechanical properties. These crystals should preferably exhibit a fine-grained formation, the structure then having the appearance of a felted network of needles. The methods by which the desired body structure can be produced and existing porcelains improved are discussed in broad outline. The quality of the matrix is affected by the kind of felspar used, its varying content of potassium, sodium, lime, silica, etc. The addition of small amounts of lime, magnesia, zinc oxide, etc. reduces the viscosity of the matrix and promotes the formation of large mullite crystals, thus increasing the translucency of the fired body, but also increasing the coefficient of expansion and hence reducing the resistance to abrupt changes of temperature. In general, the quartz in the body should be visible under the microscope only as small grains. To achieve this, the raw quartz must be ground as fine as possible and the ware must be fired for a long time at a high temperature. The above additions may also be made to accelerate the solution of the quartz in the matrix. The degree of mullite development is influenced by the type of kaolin used; relatively coarse-grained types, *e.g.*, English china clay, tend to promote the formation of large mullite crystals and thus produce more translucent bodies than the fine-grained, plastic varieties. Mullite has been identified in bodies fired to 1200°, but it develops rapidly with rising temperature. F. Salt.

Translucency of porcelain. R. Rieke and K. Samson (Ber. Deuts. Keram. Ges., 1925, 6, 189—201).—The translucency of nineteen porcelains, varying in composition between the limits of 40—60% of clay substance, 15—35% of quartz, and 15—35% of felspar, was determined with a photometer (cf. Steger, Ber. Deuts. Keram. Ges., 1921, 2, 9). The refractive index of the matrix of hard porcelain is 1·48, of mullite crystals 1·64, and of quartz crystals 1·54. Hence, mullite has a greater effect upon translucency than quartz. In general, the translucency of porcelain is increased by raising the firing temperature, and by increasing the felspar content at the expense of the quartz, the clay-substance content remaining constant. The highest translucency values were obtained with bodies containing 40—50% of clay substance, 15—30% of quartz, and 30—35% of felspar. Repeated firing to the same temperature reduced the translucency by increasing the mullite formation. The well-known Seger porcelain (low-clay-substance content) was much more translucent when fired to cone 12 instead of cone 10. Bodies prepared with kaolins of different origin exhibited marked variations in translucency; *e.g.*, the substitution of china clay for Sedlitz kaolin improved the translucency considerably. Varying the type of raw quartz used also affected the translucency. Best results were obtained with Norwegian vein quartz, which was converted almost entirely into cristobalite after one firing in a porcelain oven at cone 15. The more finely ground the quartz, the better the translucency. The effect of pegmatite, when substituted for felspar and sand, varied considerably, some varieties giving improved results, others the reverse. The addition of small amounts of lime or magnesia increased the translucency; iron oxide had the opposite effect. Preliminary fusion of the felspar with the quartz proved very effective; by fritting together these materials, so that all the quartz was dissolved, and using the resulting frit to prepare porcelain bodies with Sedlitz kaolin and English china clay, the translucency figures were 312 and 370 respectively (Seger porcelain 230).

F. Salt.

Impurities [discolorations] in fired porcelain. Hackeloer-Köbbinghoff (Ber. Deuts. Keram. Ges., 1925, 6, 236—242).—The causes to which discolorations in fired porcelain are due are discussed. Pyrites is frequently present in kaolins having a high percentage of sand (*i.e.*, insufficiently washed). The composition of the original rock and the mode of kaolinisation largely determine whether, and in what form, pyrites is present. Felspar may contain micaceous impurities; iron-containing biotite and chloride micas in particular must be carefully eliminated. Water used in grinding, or in the making operations, if not fresh, may be contaminated with filamentous algæ, minute iron-secreting organisms, the cells of which cause " iron spots " on the fired ware. Rust may be introduced into the body from

iron piping, whether galvanised or not. Grinding pans should be lined, and the lining cemented, with material containing only traces of iron. German "Findlings" quartzite is not suitable. Excessive weathering, or ageing, of porcelain body mixtures also leads to the rapid development of filamentous algæ. Compressed air, used for cleaning biscuit ware, may absorb moisture contaminated with iron from the pumps. In the firing of body mixtures which have been insufficiently ground, deposition of carbonaceous matter frequently occurs during the reducing period. F. SALT.

Elutriation process for electro-corundum. H. VIERHELLER (Sprechsaal, 1926, **59**, 113—114, 128—130; Chem. Zentr., 1926, I., 2957).—The addition of 0·04% of sulphite-cellulose waste lye of 33—35° B. (*d* 1·3—1·32) to water prevents the flocculation of the corundum, and enables the elutriation of corundum dust finer than 0·09 mm. to be carried out (cf. G.P. 399,725 and 414,909; B., 1924, 1014).

Microscopical investigation of opacity phenomena in enamel frits. A. OTREMBA (Keram. Runds., 1926, **34**, 67—70, 88—90, 107—109).—The opacifying effects of fluorine compounds in enamels, without the addition of tin oxide, were studied microscopically. Frits varying widely in composition were melted and allowed to cool in the air—not quenched in water, as is the usual practice. Fluorine was introduced in varying proportions in the form of fluorspar, natural and artificial cryolite, and sodium silicofluoride. The frits were heated to 740° and 950°, in order to study the effect of temperature. To produce an opacifying effect, fluorspar required a high temperature, or, alternatively, the use of a frit containing a high percentage of fluxes. Its opacifying effect is limited; when present in large proportion, it raises the melting point of the frit. The opacity produced is probably due to the formation of the fluorides of aluminium and of the alkalis. Natural cryolite produced greater opacity at the lower temperature than did fluorspar at the higher, the effect in this instance being due in part to the presence of minute bubbles formed by the volatilisation of fluorine or silicon fluoride. Artificial cryolite proved to be a more effective opacifier than natural cryolite. In some cases, sodium silicofluoride produced a more uniform and greater opacity at low temperatures than artificial cryolite, provided that enough felspar was present to supply the necessary alumina and alkalis. F. SALT.

Determination of porosity of refractory bricks. ESSER and PIVOVARSKY.—See II.

PATENTS.

Continuous [glass] tank furnace. E. T. FERNGREN, Assr. to LIBBEY-OWENS SHEET GLASS Co. (U.S.P. 1,581,338, 20.4.26. Appl., 27.10.23).—A furnace for supplying molten glass to a pair of glass-working machines consists of a melting tank connected to a pair of parallel refining tanks by passages narrower than the refining tanks. The enclosed heating space above each refining tank overlaps the outer sides of the tanks, and is supplied with heat from a chamber above the melting tank, so that a greater portion of the heat is directed to the outer portions of the tank. B. W. CLARKE.

Crucible and like furnaces. T. TEISEN (E.P. 238,562, 14.8.25. Conv., 14.8.24. Addn. to 194,819).—In crucible furnaces, *e.g.*, for glass, of the type described in the chief patent (B., 1923, 460 A) each recuperator is split into two units and the glass-pit is arranged between the two units. The glass-pit may be divided by a partition which may be continued upward to divide the collecting flue for waste gases, and may even be continued through the chamber floor into the crucible chamber. If each section of the recuperator is provided with an independent damper more satisfactory working of the recuperator system is possible. C. A. KING.

Removal of iron skin from waste products of glass manufacture. A. GESNEL (F.P. 601,440, 31.7.25).—Waste products of glass manufacture are treated in a bath containing a cold dilute solution of one or more mineral acids, *e.g.*, hydrochloric, sulphuric, or nitric acid, and hydrofluoric acid or a mineral, *e.g.*, fluorspar or cryolite, yielding fluorine or hydrofluoric acid. J. S. G. THOMAS.

Manufacture of enamelled objects. M. J. DAVIDSEN (E.P. 248,514, 24.12.24).—A cement, rich in alumina and poor in silica and lime, is mixed with not more than 15% of sand or Portland cement, or both, sufficient water for setting being added. Objects made from the mixtures are allowed to harden in air or water and subsequently enamelled and fired in the usual way. F. SALT.

Enamelling [cadmium and zinc]. H. C. PIERCE and C. H. HUMPHRIES, Assrs. to UDYLITE PROCESS Co. (U.S.P. 1,583,006, 4.5.26. Appl., 5.6.24).—The surfaces are treated with a solution of an arsenate and then enamelled in the usual way. F. SALT.

Opacifying enamels. VER. CHEM. FABR. KREIDL, HELLER & Co. (Austr. P. 102,118, 13.5.23. Conv., 16 and 18.5.22).—To prevent an enamel bubbling when over-fired a quantity of anhydrous material rich in alumina, *e.g.*, felspar, clay, or kaolin, which has previously been heated above 1000°, is added to the opacifier during grinding, in addition to the usual quantity of clay. A. R. POWELL.

Refractory material. GROSVENOR SCIENTIFIC PRODUCTS, LTD., and B. TURNER (E.P. 250,354, 2.2.25).—A refractory material is prepared from chrome ore (most suitably Rhodesian), crushed to pass 0·1 inch mesh and suitably graded, with refined china clay and sodium or potassium silicate. Preferably, 16 pts. by weight of ore are dry-mixed with 1¼ pts. of clay and the mixture is moistened with ½ pt. of aqueous silicate ($2\,SiO_2 : 1\,Na_2O$), *d* 1·6. The mixture is pressed in a mould, then removed, and fired. A. COUSEN.

Refractory article. A. J. JACKMAN, Assr. to VESUVIUS CRUCIBLE Co. (U.S.P. 1,577,124, 16.3.26. Appl., 2.8.24).—Articles made chiefly of graphite

and a vitrifiable ceramic bond are fired at a temperature above the vitrification point of the bonding material. F. Salt.

Tunnel kiln. H. W. Weber and A. O. Schleiffarth, Assrs. to Russell Engineering Co. (U.S.P. 1,575,470, 2.3.26. Appl., 27.11.25).—Hot air and gases in the preheating zone are passed through an inlet into a flue arranged transversely across the top wall. A fan at one end induces longitudinal circulation in the flue and discharges the hot gases into a vertical flue in the side wall, whence they are returned to the lower portion of the preheating zone. F. Salt.

Continuously-operated kiln. C. Smith (U.S.P. 1,578,559, 30.3.26. Appl., 23.2.23).—A kiln for burning and drying brick and the like consists of a vertical drying shaft, up which the material is conveyed, communicating near the top by means of a flue with a vertical burning shaft connected with a fire-box. B. W. Clarke.

Ceramic material. R. T. Stull, Assr. to Stull Process Co. (U.S.P. 1,576,558, 16.3.26. Appl., 20.3.24).—Light-coloured bricks are made from a mixture composed of a natural, highly colloidal, secondary kaolin, deficient in alkali and alkaline-earth fluxes, and a pegmatite. F. Salt.

Cast or moulded articles [of titanium-containing slag]. F. A. de Silva (U.S.P. 1,579,093, 30.3.26. Appl., 9.4.25).—The molten slag obtained in the direct production in the electric furnace of titanium steel from titaniferous iron ores contains principally titanic acid and titanium carbide and when cooled, particularly if the cooling takes place in moulds under pressure, forms an extremely hard, resistant and practically infusible substance suitable for use as a lining for furnaces, in the manufacture of crucibles, etc. For roofing purposes and the like it is coated on asbestos when molten. T. S. Wheeler.

Artificial aluminous abrasive. H. A. Richmond and R. Macdonald, jun., Assrs. to Gen. Abrasive Co. (U.S.P. 1,583,179, 4.5.26. Appl., 9.8.24).—The operating temperature in the production of crystalline alumina from an aluminous ore is lowered by the addition of an alkaline-earth to the furnace charge. F. Salt.

Refractory brick for furnace linings (U.S.P. 1,576,021).—See I.

IX.—BUILDING MATERIALS.

Lime-silica index as measure of cement quality. T. Merriman (Eng. News. Rec., 1926, 96, 648—650).—The lime-silica index is the ratio of the available lime, *i.e.*, the total lime less that in combination with the alumina, ferric oxide, and sulphuric acid, to the available silica, *i.e.*, total silica less insoluble residue. Assuming that tricalcium silicate is the most desirable constituent of Portland cement, a high index should indicate a cement of good quality. Actually, owing to incompleteness of combination of the calcium silicates during manufacture, a high lime-silica index is accompanied by a relatively low strength at 7 days. This shows that the burning operation has been badly carried out and that a large proportion of inert or underburned material is present. In order to ensure that the maximum cementing value is obtained, it is necessary to specify both the strength and the lime-silica index of the cement. B. W. Clarke.

Rapid determination of lime in cement and raw materials for cement. H. Kühl and F. Klasse (Zement, 1926, 15, 181—184; Chem. Zentr., 1926, I., 2958).—0·3 g. of the ignited material is treated with 5 c.c. of fuming nitric acid on the water bath, and after evaporating the excess of nitric acid, an excess of powdered oxalic acid crystals is added and the mixture heated gradually in an electric furnace, finally keeping at 500—550° for 20—30 min. After cooling, the material is transferred to a calcimeter. B. W. Clarke.

Fineness of particles of cement, especially iron Portland cement. A. Guttmann (Zement, 1926, 15, 164—168, 185—187, 200—203; Chem. Zentr., 1926, I., 2955).—The finest particles in cement have generally a higher percentage of lime and gypsum than the larger particles. Of two iron Portland cements, the one which was the finer by sieve analysis, showed the lower strength, but use of a 10,000-mesh sieve showed that the other had the larger proportion of very fine particles. Sedimentation in the Bauer apparatus is necessary for the quantitative determination of the finest particles. B. W. Clarke.

Endurance of flooring materials. C. H. Geister (J. Amer. Ceram. Soc., 1926, 9, 126—130). —The durability and the sanitary properties of all types of flooring materials were investigated on the basis of the following tests: resistance to abrasion, absorption of water, indentation, resistance to chemicals and stains, and deterioration from age. The results of the tests, which are tabulated, show clearly the superiority of ceramic tiles over other materials. F. Salt.

Patents.

Manufacture of aluminous cements, coloured or white. U. B. Voisin (E.P. 248,282, 14.10.25. Addn. to 243,876; B., 1926, 129).—The aluminous cement mass, very finely ground and very intimately mixed, is roasted for a long period (6 to 8 hrs.) at a temperature well below the softening point (1000—1100°). White cement is produced by the use of white bauxite and pure lime. F. Salt.

High-temperature cement. P. G. Willetts, Assr. to Hartford-Empire Co. (U.S.P. 1,573,888, 23.2.26. Appl., 13.11.25).—A refractory cement is prepared from barium sulphate, silica, and sodium silicate. F. Salt.

Manufacturing cements [plaster] from overburnt gypsum, overburnt gypsum waste, or

natural anhydrite. P. P. Budnikov and M. E. Lewin (G.P. 320,957, 26.1.24).—A hard-setting plaster is produced, without the necessity of employing any simultaneous or subsequent heat treatment, by mixing the raw material intimately with the appropriate amount of acid alkali sulphate, *e.g.*, sodium hydrogen sulphate or potassium hydrogen sulphate, or neutral alkali salt together with the requisite amount of sulphuric acid. J. S. G. Thomas.

Induration or minerlisation of organic matter. C. D. Burney (E.P. 249,899, 25.10.24).—Fibrous organic material, either moistened, or washed with a weak solution of an alkali or of an acid, is treated with a dry hygroscopic chemical which reacts exothermically with water, such as dry caustic soda preferably in flakes, or dry caustic lime, or magnesium chloride, and mixed, while heat is still being evolved, with cement, mortar, and the like to produce on setting a light form of concrete which can be moulded under pressure. B. W. Clarke.

Coloured granulated slag and method of manufacture. W. Van de Mark, Assr. to Vulcan Louisville Smelting Co. (U.S.P. 1,582,318, 27.4.26. Appl., 23.5.24. Renewed 25.2.26).—A granulated material, suitable for covering roofing felt, is obtained by smelting the residue from brass or copper smelting furnaces with a calcium compound to reduce its iron content, and pouring into water. A. Geake.

Preservation of stone. A. P. Laurie (U.S.P. 1,585,103, 18.5.26. Appl., 16.8.24).—See E.P. 221,342; B., 1924, 911.

Rotary drums for cooling materials (E.P. 250,318).—See I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Testing cast-iron [previous to casting]. P. Wolff (Stahl u. Eisen, 1926, **46**, 560—564).—Before casting a charge of cast-iron a small wedge-shaped casting should be made in a sand mould. The Brinell hardness of this casting after removing the outer layer gives sufficiently accurate indications of the quality of a charge once it has been standardised against analyses and tensile tests on similar material. The fracture of the test-piece also provides information as to the probable structure of the castings that will be obtained. The sharp end of the wedge will indicate the depth of the white hard layer that may be expected and the upper end of the wedge the nature of the grey interior of the casting. The tensile strength of the metal is given approximately by the equation, $T=(H-40)/6$, where H is the Brinell hardness. A. R. Powell.

Influence of prolonged heating on the crystallisation of combined carbon in cast-iron. O. Wedemeyer (Stahl u. Eisen, 1926, **46**, 557—560).—A series of large-scale tests in a coal-fired reverberatory furnace has confirmed Pivovarski's observation (B., 1925, 805) that prolonged heating at temperatures below 1500° increases the carbide content of cast iron. The increase is much more marked in the case of cast-iron made from soft iron than in that made from hæmatite iron; thus, overheating for 3 hrs. at 1295° increased the combined carbon in the first case from 1·12 to 2·57% and in the second case only from 0·8 to 1·00%. A. R. Powell.

Behaviour of aluminium towards iron at high temperatures. R. Irmann (Z. Metallk., 1926, **18**, 121—122).—The amount of iron dissolved by molten aluminium increases with rise of temperature but depends on the nature of the iron, a high silicon and/or carbon content decreasing the solubility of the iron. Thus, aluminium at 800° dissolves in 1 hr. 1% of iron from a steel containing 0·12% C, but none from grey cast-iron with or without the casting skin; at 900°, 2·5% of iron is dissolved from the steel, 0·5% from cast-iron with the skin removed, and nothing from cast-iron with the skin, and at 1000° the amounts dissolved are 6%, 2·5%, and 1% respectively. The solubility of a cast-iron in aluminium decreases hyperbolically with the content of combined carbon and linearly with the silicon content. Cast-iron crucibles are therefore the most suitable for melting aluminium especially if the inner surface is coated with aluminium and then heated to 1000° before use. Steel crucibles similarly treated do not give such good results, but both cast-iron and steel crucibles that have been heated until a thin layer of ferrosoferric oxide is formed inside yield only traces of iron to aluminium that is melted in them. A. R. Powell.

Tensile properties of metals [steel] at high temperatures. T. D. Lynch, N. L. Mochel, and P. G. McVetty (Proc. Amer. Soc. Testing Materials, 1925, **25**, II, 5—26; Chem. Abstr., 1926, 20, 1202).—The elastic properties of medium-carbon steel decrease with rise of temperature (up to 500°), the ductility is minimal at about 250°, whilst the tensile strength is minimal at 100° and maximal at 275°. Nickel steel (5% Ni) was similar, but exhibited better ductility. "Stainless" iron showed much less effect with rise of temperature. Cast manganese bronze showed no change in elastic properties up to 260°, but a drop in tensile strength. Previous heat treatment of the medium-carbon steel reduced the final rate of extension under stress; manganese bronze elongated more than steel in the long-time tests. At a constant high temperature the stress varied linearly with the logarithm of the time to cause fracture. Medium-carbon steel stressed above the proportional limit for 200 hrs. at 400° showed at the normal temperature decreased ductility, increased strength, and increased elastic properties, except the modulus. A. A. Eldridge.

Elastic limit and the cold and hot shortness [of steel]. P. Ludwik (Z. Ver. Deuts. Ing., 1926, **70**, 379—386; Chem. Zentr., 1926, I., 3176—3177).—The changes in the elastic limit of electrolytic iron and ingot steel during ageing and at a "blue heat," as well as the "blue brittleness" and other phenomena of the hardening of steel, are shown to be due to changes in the solubility of the minor consti-

tuents in the steel just as is the case with the hardening of aluminium alloys during ageing. The cold brittleness, the dependence of the notched bar impact test figures on the shape of the notch, the velocity of the blow, and the temperature and the possibility of rupture before the beginning of elastic flow in tensile and impact tests are caused by changes in the slip and shear resistance of the metal. The shear strength of steel is decreased during pickling owing to adsorption of hydrogen in the surface layers of the metal, but this has no effect on the elastic limit or tensile strength. The brittleness induced in steel by contact with alkali hydroxide solution is caused by a reduction in the resistance to shear caused by hydrogen adsorption, and an increase in the slip resistance caused by ageing.

A. R. Powell.

Moduli of elasticity and rigidity and their change caused by magnetisation in different kinds of steel. K. Honda and T. Tanaka (Sci. Rep. Tôhoku, 1926, **15**, 1—37).—The moduli of elasticity and of rigidity of carbon steels decrease with an increase of the carbon content and also after annealing and quenching, but they are increased after magnetisation of the steel. The elastic constants of cobalt–iron alloys are increased by magnetising except in the case of alloys containing 35—55% Co, the elastic constants of which decrease in weak magnetic fields. Similar changes take place on magnetising nickel–iron alloys. The elastic moduli of cobalt are reduced by magnetisation. In all cases the effect of a magnetic flux is three times as great on the rigidity modulus as on the elastic modulus.

A. R. Powell.

Properties of high-silicon structural steels. (Stahl u. Eisen, 1926, **46**, 493—503).—Tests carried out on low-carbon steels containing about 1% Si, manufactured at various foundries in different types of furnaces and under different rolling conditions, confirm previous work on the influence of silicon. The tensile strength and, in higher degree, the yield point, properties markedly affected by the shape of the test-piece, are increased without diminution of the percentage elongation and reduction in area of the test–piece.

L. M. Clark.

Primary crystallites in chrome–nickel steels. F. Leitner (Stahl u. Eisen, 1926, **46**, 525—533).—Specimens of chrome–nickel steels cast at different temperatures were etched to show the structure, and mechanical tests were made. The formation of large crystallites is disadvantageous, leading to local accumulation of impurity and formation of holes with corresponding injury to mechanical properties. These properties can be improved by suitable heat treatment.

L. M. Clark.

Mechanical properties of titanium steel. K. Tamaru (Sci. Rep. Tôhoku, 1926, **15**, 73—80; cf. B., 1925, 593).—Addition of 0·3% Ti to plain carbon steels increases the tensile strength and reduction in area, but decreases the elongation. Owing to the tendency to the formation of titanium nitride when the steels are melted in air, better mechanical properties are obtained by melting *in vacuo* or under a low pressure of hydrogen. The yield point of titanium steel decreases with an increase in the carbon content whereas the elongation is hardly affected.

A. R. Powell.

Cementation of ferrous and cuprous alloys by tungsten, molybdenum, and tantalum. J. Laissus (Compt. rend., 1926, **182**, 1152—1154; cf. B., 1925, 635; 1926, 278).—The cementation of an iron–carbon alloy containing 0·15% of carbon, by finely powdered ferromolybdenum (C 1·86% Mo 71·85%) or ferrotantalum (C 1·00%, Ta 29·26%, Si 1·96%) has been studied between 800 and 1200°. Micrographical examination shows the existence in the interior of a solid solution bounded by a brilliant external layer, the thickness of which increases with rise of temperature and with the duration of treatment. Copper and brass have been similarly treated with tungsten, molybdenum, and tantalum; cemented layers are formed which increase in thickness with rise of temperature.

F. G. Soper.

Corrosion of steels in the atmosphere. W. G. Whitman and E. L. Chappell (Ind. Eng. Chem., 1926, **18**, 533—535).—Test specimens enclosed in a galvanised box were sprayed intermittently with water for periods of 15 min. at intervals ranging from 30 min. to 24 hrs., the cycle of operation being regulated automatically. The rate of corrosion compared well with that of specimens in actual field tests. The rate of corrosion varied with the time of the cycle of operation, and appeared to depend on the actual time the specimen was wetted. With chromium steels, the corrosion decreased rapidly as the chromium increased from 8 to 16%.

B. W. Clarke.

Resistance to corrosion of Thomas and Siemens–Martin steels containing copper. K. Daeves (Stahl u. Eisen, 1926, **46**, 609—611).—The differences in the conclusions arrived at by the Amer. Soc. for Testing Materials and those of O. Bauer (cf. B., 1921, 393 A) from data collected by the Verein deutscher Eisenhüttenleute and the Materialprüfungsamt in Berlin-Dahlem, are discussed. To test whether these were due to climatic conditions or to fundamental differences between German and American steels, experiments have been carried out in which ten specimens of each of various kinds of steel containing varying amounts of copper up to 0·5% have been exposed to the air without regard to the district, and the amount of corrosion determined by weighing the plates before exposure and again after removing the rust. The results are expressed in curves in which the loss in weight is plotted against the % of copper in the steel. In agreement with American results, amounts of copper up to 0·25% are found very effective in retarding corrosion in all the classes of steel tested. The improvement is most marked in the case of Thomas steel. Siemens-Martin steel made by the crude ore process occupies a middle position but Siegerlander material (high natural copper content) is almost as good as Thomas steel. The influence of copper when the steel is

exposed to earth corrosion is not so marked, but again Thomas steel is the more resistant.
A. COULTHARD.

Dilatometric anomaly of paramagnetic nickel–chromium alloys. Alloy for dilatation pyrometry. P. CHEVENARD (Compt. rend., 1926, **182**, 1281—1283; cf. B., 1925, 674).—Industrial nickel–chromium alloys (5—10% Cr, 2% Mn) exhibit an anomaly in the dilatation curve near 550°, which is imperfectly reversible. The anomaly is almost completely suppressed in a new alloy ("pyros") in which the manganese content has been increased and iron and tungsten added (82% Ni, 7% Cr, 5% W, 3% Mn, 3% Fe). An expression for the dilatation of "pyros," with the constants determined, is given.
R. A. MORTON.

Surface films in the cathodic polarisation of metals. E. LIEBREICH (Korrosion u. Metallschutz, 1926, **2**, 38—41; Chem. Zentr., 1926, I., 2965—2966).—As is the case with chromium, iron, and mercury, there is a potential range in the feeble cathodic polarisation of nickel and aluminium in which the metal has a much greater tendency to dissolve in the electrolyte with the formation of surface films of basic salts. A similar phenomenon appears to take place during the slow natural corrosion of these metals, and is probably the cause of the feeble alkalinity observed by Wurstemberger in the liquid immediately in contact with a corroded metal surface. The facts are explained as follows: during cathodic polarisation the charged hydrogen first formed at the cathode is adsorbed either by the metal or by the solution, leaving a film of solution containing an excess of hydroxyl ions which react with the metal to form basic salts or hydroxide; as the polarisation increases the increasing quantities of hydrogen produced reduce these salts again to metal, after which the hydrogen is evolved in bubbles. A. R. POWELL.

Rapid determination of small quantities of palladium. F. C. ROBINSON (Inst. Min. Met., May, 1926. Advance copy, 3 pp.).—Small quantities of palladium may be determined colorimetrically in the acid used in parting the gold–silver–platinum–palladium bead obtained by cupellation. A metal ratio of 33 Ag : 10 Au : 1 (Pt+Pd) is necessary for satisfactory parting of the bead and, in cases where nitric acid is used, the platinum should not exceed 10 times the weight of the palladium, and in no case more than 50 mg. per 100 c.c. of acid. Sulphuric acid answers equally well for the parting and has the advantage that it does not dissolve platinum. The standard palladium solution contains 1 mg. Pd per c.c., and is made by dissolving the requisite amount of metal in 1 : 1 nitric acid, and evaporating with sulphuric acid in case a sulphate solution is required.
A. R. POWELL.

Production of a regular structure in the recrystallisation of [cold-rolled] copper. W. KÖSTER (Z. Metallk., 1926, **18**, 112—116).—When severely cold-worked copper is annealed the new crystals are oriented in such a way that the twinning planes are at an angle of 45° to the direction of rolling and deep etching shows that the new crystals have a cube face in the plane of rolling and a normal to the cube in the direction of rolling. The recrystallised grains are extended further in the direction of rolling than in the direction perpendicular to it. The regularity of the recrystallised structure decreases with the amount of cold work to which the metal was previously subjected, but the orientation of the new structure is independent of the annealing temperature, time of annealing, and rate of heating. The tensile strength, ductility, and pliability of recrystallised sheet are greatest at an angle of 45° to the direction of rolling, and the tensile strength is at a minimum at an angle of 22·5° to the direction of rolling.
A. R. POWELL.

Determination of aluminium oxide in aluminium metal. W. H. WITHEY and H. E. MILLAR (J.S.C.I., 1926, **45**, 170—174 T).—The only method found satisfactory consisted in heating the metal in dry gaseous hydrogen chloride, and determining the residual oxide. Experiments made on foil of various thicknesses indicate that the oxide is confined to a minute surface layer, but that the proportion of oxide in thin foil is not, as might be expected, larger than on thick sheet. By reducing oxides of lead and iron by fusion with molten aluminium and analysis of the resulting ingots cast from the skimmed metal, it is shown that the alumina produced during the reaction, although formed under most favourable conditions for solution in the metal, is almost completely expelled; in other words, molten aluminium is incapable of dissolving its oxide and any oxide originally present in the metal is probably confined to a minute surface layer. A method of determining carbon in aluminium by dissolving the metal in a mixture of sulphuric and hydrochloric acids and burning the evolved gases is described.

Complexity of the phenomena of quenching of certain alloys. A. PORTEVIN and P. CHEVENARD (Compt. rend., 1926, **182**, 1143—1145; cf. A., 1921, ii., 510; 1923, ii., 166).—Two general cases are distinguished in the quenching of alloys. In the first the alloy has a γ state stable at high temperatures, and an α state stable in the cold; any treatment which refines the structure of α increases the hardness of the alloy. In the second case, there exists in addition a state λ in labile equilibrium with the γ state. Evidence for this view is adduced from a dilatometric study of an aluminium-bronze, sudden cooling causing the formation of the λ state which is attended by an expansion. F. G. SOPER.

Tensile strength of metals and alloys under a statical load. G. WELTER (Z. Metallk., 1926, **18**, 75—80, 117—120).—Rods of different cross-sections of brass, copper, aluminium, and steel were subjected to a load above the elastic limit, but below the breaking strain, for prolonged periods until fracture ensued. Coarse-grained $\alpha+\beta$-brass withstood loads up to within a few kg. per sq. mm. of the breaking strain almost indefinitely, but little more than the load required to stretch the metal beyond the elastic limit sufficed to cause fracture of fine-grained brass

of similar composition within a few hours or days. Copper and aluminium failed in the course of several months under loads just above the limit of proportionality, whereas steel withstood loads almost equal to the tensile strength indefinitely. Round bars in all cases were stronger than notched bars and bars with a rectangular notch were stronger than those with a wedge-shaped notch. The importance of these results in testing metals for constructional work is pointed out. A. R. Powell.

Fatigue of metals by direct stress. P. L. Irwin (Proc. Amer. Soc. Testing Materials, 1925, **25**, II., 53—65; Chem. Abstr., 1926, **20**, 1203).—The endurance limit obtained for steels and for manganese bronze by direct stress is the same as that obtained by flexural stress. A. A. Eldridge.

Granulation of slags and metals. B. Bogitch (Compt. rend., 1926, **182**, 1221—1223).—In the granulation of slags and metals by quenching, dangerous explosions may occur if too much molten metal is entrained in the molten slag. A jet of compressed air introduced below the water surface serves to retard and break up the molten stream, and to prevent the formation of a solid sheet over the surface of the vessel. Experiments with an iron–magnesium silicate slag and a molten metal, using air compressed to 1 kg., showed that quenching may safely be carried out in smaller vessels, and from a less height than is usual. The hot water produced may also be utilised. J. Grant.

Rôle of sodium sulphate in the refining of mattes. B. Bogitch (Rev. Mét., 1926, **23**, 193—198).—See B., 1925, 995.

See also A., May, 486, **Action of brass on cuprous chloride** (Masing and Miething); **Gold in mercury** (Venator). 488, **Reaction between manganese and iron sulphide** (Herty, jun., and True). 489, **Preparation and properties of metallic yttrium** (Thompson, Holton, and Kremers). 490, **Precipitation of tellurium from alkaline sulphide solutions and its separation from heavy metals and from selenium** (Brukl and Maxymowicz). 491, **Determination of potassium** (Delaville and Carlier; Wikul; Le Boucher); **Determination of small amounts of silver in presence of lead** (Donath); **"Citarin" as a quantitative reagent** (Vanino and Guyot); **Determination of calcium** (Foote and Bradley); **Compounds of diphenylthiocarbazone with metals and their use in analysis** (Fischer); **Determination of cadmium in copper** (Blazey). 492, **Determination of manganese, zinc, and cobalt** (Strebinger and Pollak); **Volumetric determination of chromium and manganese** (Reinitzer and Conrath).

See also pages 522, **Determination of porosity of rammed moulding sand** (Esser and Pivovarski). 549, **High-frequency induction furnaces** (Wever); **Flames of atomic hydrogen** (Langmuir). 550, **Arc welding with atomic hydrogen** (Weiman and Langmuir); **Arc welding in hydrogen and other gases** (Alexander); **Influence of cathodic hydrogen on strength of steel** (Alexejev and Polukarov).

Patents.

Electrodepositing iron from minerals. M. Okochi and M. Hanaoka, Assrs. to Zaidan Hojin Rikagaku Kenkyujo (U.S.P. 1,582,423, 27.4.26. Appl., 2.4.24).—Iron oxide ores, such as magnetite sands, yellow ochre, and bog iron ore, are reduced to spongy iron at 1000°, and a mixture of this iron and material containing a sulphide of iron is made in the anode in an acid electrolyte. A. R. Powell.

Manufacture of acid-resistant iron–silicon alloys. H. Fischer (G.P. 426,113, 26.11.19).—Copper is added to the alloys to lower their content of silicon. A. Coulthard.

Casting aluminium and aluminium alloys and moulds for use therein. E. Strasser (E.P. 244,441, 18.11.25. Conv., 13.12.24).—Sand moulds for casting aluminium alloys are made of a mixture of "green" sand and a metal or metal compound, such as copper filings or roasted copper sulphide ores. The moulds are used damp so as to ensure as rapid a chilling of the casting as possible. Aluminium–zinc alloys cast in this manner and aged for 14 days show superior mechanical properties to similar alloys cast in the ordinary sand moulds and heat-treated. A. R. Powell.

Separation and purification of platinum and other platinum metals from platiniferous ores and substances. L. D. Hooper (E.P. 250,726, 6.3.25).—Platinum is removed from platiniferous materials by treating them with carbon monoxide and chlorine at 250—600° in the presence of a catalyst such as metallic sodium, whereby carbonyl compounds of platinous chloride are volatilised. These may be purified by recrystallisation from carbon tetrachloride. Palladium is recovered from the residue from the platinum extraction by treatment with chlorine and carbon monoxide at 300—650° in the presence of ammonium fluoride or chloride or of hydrogen chloride, whereby similar palladous chloride carbonyl compounds are volatilised. By subjecting the residue from this treatment to carbon monoxide at 300° under 400 atm. pressure ruthenium dicarbonyl is formed and may be extracted with alcohol from the remainder of the mass. The metals are recovered from the carbonyl compounds by heating, with or without the presence of water. A. R. Powell.

Refining copper–nickel matte. N. V. Hybinette, Assr. to Anglo-Canadian Mining and Refining Co., Ltd. (U.S.P. 1,577,422, 16.3.26. Appl., 13.1.21).—A solution of copper and nickel sulphates, obtained from a later stage of the process, is treated with bessemerised copper–nickel matte to obtain a purified nickel sulphate solution for electrolysis, and a mixture of matte and cement copper which is roasted to remove sulphur. The roasted product is leached with sulphuric acid to obtain copper sulphate solution which is electrolysed for copper and the regeneration of sulphuric acid for further leaching of roasted matte. The nickeliferous residues from leaching are reduced and

dissolved in sulphuric acid regenerated during the previous nickel electrolysis and the solution is treated with a fresh amount of matte as before.
A. R. POWELL.

Electrolytic precipitation of copper. F. LAIST and F. F. FRICK, Assrs. to ANACONDA COPPER MINING CO. (U.S.P. 1,580,614, 13.4.26. Appl., 23.10.25).—In a process involving the leaching of the ore with an acid solution, the electrolysis of the leaching solution to precipitate the copper and regenerate the acid, and the use of this acid for leaching more ore, the leaching solution is also treated with a reagent capable of precipitating iron.
H. HOLMES.

Extraction of copper from ores and the like. F. M. MURDOCH (Austral. P. 16,878, 14.3.24).—Finely ground copper ores, concentrates, or residues are roasted, if necessary, and leached with a solution of calcium, magnesium, or ferric chloride containing sodium chloride, while a current of sulphur dioxide is passed through the mixture. After filtration copper is recovered from the solution by precipitation with calcium or magnesium hydroxide.
A. R. POWELL.

Precipitation of copper from impure solutions of copper sulphate by electrolysis. G. BOSSIÈRE (G.P. 426,448, 30.8.24. Conv., 4.3.24).—The electrolysis is carried out in two stages: in the first, when the copper content of the solution is high, pure copper is deposited at the cathode; the second, with the solution weak in copper, is carried out in presence of sulphur dioxide, which is reduced to hydrogen sulphide at the cathode causing the precipitation of the remaining copper and other metals, such as arsenic, as sulphides.
A. COULTHARD.

[White] gold alloy. T. P. SHIELDS, Assr. to SHIELDS & MOORE (U.S.P. 1,580,443, 13.4.26. Appl., 15.5.24).—A white gold alloy contains more than 70% Au, together with more than 15% of nickel and iron, the iron content being more than half the nickel content. A small amount of a deoxidising metal which forms an oxide of the type MO_2 is added to the molten alloy before casting. A. R. POWELL.

Alloy. Gold alloy. T. P. SHIELDS, Assr. to SHIELDS & MOORE (U.S.P. 1,580,444—5, 13.4.26. Appl., [A] 20.5.25 and [B] 6.7.25).—An intermediate alloy for use in making gold alloys consists of (A) 1—10 pts. of chromium, 24—50 pts. of copper, 10—30 pts. of zinc, and up to about 15 pts. of silver, or (B) 0·1—2 pts. of chromium, 24—50 pts. of copper, and 10—18 pts. of zinc. A. R. POWELL.

Electrodepositing chromium and preparing baths therefor. C. G. FINK, Assr. to CHEMICAL TREATMENT CO., INC. (U.S.P. 1,581,188, 20.4.26. Appl., 19.12.25).—Chromium is electrodeposited from solutions of chromic acid by reducing the chromic acid at the cathode in the presence of a catalyst and of a hydrogen film. A. R. POWELL.

Making metallic chromium. W. E. S. STRONG, C. E. PARSONS, and S. PEACOCK, Assrs. to METAL RESEARCH CORP. (U.S.P. 1,581,698, 20.4.26. Appl., 19.2.25).—A mixture containing chromic oxide, a sodium compound containing oxygen, and carbon is smelted in a blast furnace in such a way that the metallic sodium first liberated reduces the chromic oxide to metallic chromium. A. R. POWELL.

Extracting arsenides from ores. J. F. SANDERS (U.S.P. 1,581,475, 20.4.26. Appl., 22.4.25).—Ores contaminated with arsenic are finely ground and agitated with carbon disulphide until the arsenic compounds are dissolved. A. R. POWELL.

Recovery of values from ores. G. D. VAN ARSDALE, H. W. ALDRICH, and W. G. SCOTT, Assrs. to INSPIRATION CONSOLIDATED COPPER CO. (U.S.P. 1,581,479, 20.4.26. Appl., 7.9.23).—In the leaching of ores the residual solids from the first percolation are washed on the counter-current principle to obtain a solution suitable to be added to a rich liquor for the usual treatment. The remaining values are recovered by circulating liquid through the residues and removing the values from the solution after each washing by any method which permits of almost complete recovery. A. R. POWELL.

Uniting metals [with copper or its alloys]. G. D. BAGLEY, Assr. to ELECTRO-METALLURGICAL CO. (U.S.P. 1,582,024, 27.4.26. Appl., 29.5.26).—Metals of the chromium group may be joined to copper by interposing a nickel layer between the two metals and causing one surface of the nickel to alloy with the chromium group metal and the other surface with the copper. A. R. POWELL.

Reducing metal sulphides. J. W. BECKMAN, Assr. to BECKMAN-LINDEN ENGINEERING CORP. (U.S.P. 1,582,157, 27.4.26. Appl., 22.5.22).—Metal sulphides may be reduced to the corresponding metal by fusing them with a mixture of a basic oxide, a flux, and carbon. A. R. POWELL.

Method of operating blast furnaces. R. D. LANCE (F.P. 601,159, 20.10.24).—The process consists in replacing a part or the whole of the solid fuel by gaseous fuel and the enrichment of the furnace gases as they enter the recuperators with a suitable fuel gas. C. T. GIMINGHAM.

Increasing the oxygen content of air blasts for use in metallurgical processes. A. WAGNER and K. THOMAS (G.P. 426,738, 20.9.22).—The proportion of oxygen is increased by passing the air through porous tubes, through which the nitrogen diffuses more rapidly than the oxygen. L. A. COLES.

Manufacture of metals and alloys in the electric furnace. E. A. A. GRÖNWALL (F.P. 601,350, 27.7.25).—The process involves the use of one or more vertical electrodes and the introduction of the material to be heated, in a finely divided form, through openings in the walls near the lower end of the electrodes. C. T. GIMINGHAM.

Smelting metals and ores. Soc. des Prod. Métallurgiques Constant-Bruzac (F.P. 601,187, 23.10.24).—The carbon dioxide produced by combustion of the fuel is allowed to act on the material to be smelted. A. Coulthard.

Chill-cast alloy resistant to the action of acids and alkalis. R. Siller (G.P. 426,835, 16.11.24).—Small quantities of molybdenum, tungsten, nickel, and cobalt are added to the mixture for producing the alloy, the proportions of the molybdenum and tungsten, and of the cobalt, exceeding that of the nickel. L. A. Coles.

Hard alloys for use in the manufacture of tools. Siemens & Halske A.-G., Assees. of B. Fetkenheuer (G.P. 427,074, 25.3.22).—The alloys are obtained by adding tungsten carbide to molten metals or alloys; *e.g.*, 1—20% is added to a molten alloy containing 1 pt. of chromium and 1—2 pts. of cobalt. L. A. Coles.

Hard alloys which contain silicon and carbon in addition to other substances. Siemens & Halske A.-G., Assees. of B. Fetkenheuer (Austr. P. 102,286, 18.6.24. Conv., 18.6.23).—Rods containing silicon carbide are dipped into the melt, and heating of the melt is continued until the desired amounts of silicon and carbon have been taken up. A. Coulthard.

Silver–copper alloys of high silver content. G. A. Scheid'sche Affinerie (Austr. P. 102,287, 28.7.24).—The alloys contain silicon or tin or tin-silicon or silicon-bronze to produce the required grade of malleability. A. Coulthard.

Smelting of ores of antimony, arsenic, and mercury. Oesterr. Bamag-Büttner-Werke A.-G., and R. Jahn (Austr. P. 102,293, 11.2.25).—The ground ores are roasted by external heating and the volatilised metal oxides passed through a dust settling chamber kept at a temperature above their precipitation point. The purified vapours are then led to a reduction flame, and the metals so produced are separated by fractional cooling. A. Coulthard.

Concentrating ores and other materials. E. C. R. Marks. From Ellis Flotation Co., Inc. (E.P. 251,171, 12.10.25).—See U.S.P. 1,555,915; B., 1925, 963.

Manufacturing ductile tungsten. A. Just, Assr. to General Electric Co. (U.S.P. 1,585,497, 18.5.26. Appl., 23.2.24).—See E.P. 214,662; B., 1925, 553.

Recovering vanadium compounds from iron ores containing vanadium and titanium. B. P. F. Kjellberg (U.S.P. 1,583,053, 4.5.26. Appl., 6.5.25).—See F.P. 598,315; B., 1926, 321.

Casting [easily oxidisable] metals. Chem. Fabr. Griesheim-Elektron, Assees. of A. Beck (U.S.P. 1,584,072, 11.5.26. Appl., 16.7.23).—See G.P. 384,137; B., 1924, 388.

Method of manufacturing metal powder. G. Shimadzu (U.S.P. 1,584,149, 11.5.26. Appl., 30.1.22).—See E.P. 197,031; B., 1923, 725 a.

Means for agitating and stirring molten metal in open-hearth and like furnaces. J. Edwards (E.P. 251,513, 28.9.25).

See also pages 524, **Gaseous fuel** (U.S.P. 1,581,441). 542, **Enamelling cadmium and zinc** (U.S.P. 1,583,006). 543, **Moulded articles of titanium-containing slag** (U.S.P. 1,579,093). 544, **Coloured granulated slag** (U.S.P. 1,582,318).

XI.—ELECTROTECHNICS.

High-frequency induction furnaces. F. Wever (Stahl u. Eisen, 1926, **46**, 533—536).—The energy requirement of high-frequency induction furnaces is less than that of any other type of furnace. A 35-kg. charge can be melted in 1 hr. using 48 kw. A larger choice of lining materials is made available by the increased life-period of furnace linings due to avoidance of local overheating in the process. Since no electrodes are used, charges can be melted *in vacuo* or in an oxidising or reducing atmosphere. The carbon content of alloy steels can therefore be lowered by oxidation, whilst the velocity of slag-reactions which lower the content of phosphorus and manganese is increased by the agitation of the molten charge caused by eddy-currents. L. M. Clark.

Colloid condenser. A. Nodon (Compt. rend., 1926, **182**, 1270—1272).—The colloid condenser consists of sheets of aluminium or magnesium protected by ebonite or paraffined paper. The sheets are separated by a meshed insulator such as canvas in which the interstices are filled with a thick paste of colloidal ferric oxide and glycerin. The foils can be connected to an alternating current supply of a few volts to act as an electrostatic condenser of great capacity. Twenty such couples connected in series will support 110 volts a.c. without appreciable loss. A condenser of this type weighing 100—150 g. possesses a capacity of several hundred microfarads. Colloidal oxides of nickel, chromium, and manganese act similarly. The capacity of the colloid condenser decreases rapidly with increasing surface. The arrangement has the advantage that there is little danger of destruction by internal discharges. R. A. Morton.

Flames of atomic hydrogen. I. Langmuir (Science, 1925, **62**, 463—464; Chem. Zentr., 1926, I., 1119).—Atomic hydrogen in considerable concentration is produced by a powerful arc discharge between tungsten electrodes in an atmosphere of hydrogen (cf. E.P. 237,898; B., 1926, 198). By blowing a current of hydrogen from a narrow tube into the arc, an extremely hot flame of atomic hydrogen is produced which melts molybdenum and tungsten with ease. In the presence of hydrogen no oxidation of the metal surface can occur; the

process is therefore suitable for welding (cf. E.P. 237,901; B., 1926, 368). A. B. MANNING.

Flames of atomic hydrogen. I. LANGMUIR (Gen. Electric Rev., 1926, 29, 153—159; Chem. Zentr., 1926, I., 2886; cf. preceding abstract).—The abnormally high thermal conductivity of hydrogen at high temperatures (at 3400° it is about 23 times that of nitrogen) is due to dissociation. Assuming the heat of reaction of the change $H_2 \rightarrow 2H$ to be 98,000 cal., the degrees of dissociation at 1000°, 2000°, 3000°, 4000°, and 5000° Abs. are $3{\cdot}71 \times 10^{-7}$, $1{\cdot}22 \times 10^{-1}$, 9·03, 62·3, and 94·7% respectively. Atomic hydrogen is produced by blowing a current of the gas through an arc of high current density (20 amp. at 300—800 volts) between tungsten electrodes. If the stream of gas containing atomic hydrogen is directed against a metal surface, then in consequence of the catalytic action of the surface, the reverse change, from atomic to molecular hydrogen, takes place with the liberation of 98,000 cal. per 2 g. of hydrogen. In this way it has been possible to melt the most highly refractory substances such as tungsten, tantalum, and thorium oxide, and to attain temperatures higher than those of the oxy-acetylene flame. A. B. MANNING.

Arc welding with atomic hydrogen. R. A. WEIMAN and I. LANGMUIR (Gen. Electric Rev., 1926, 29, 160—168; Chem. Zentr., 1926, I., 2886; cf. preceding abstracts).—Langmuir's atomic hydrogen flame has been applied to the welding of metals. All types of alloys can be welded without deterioration of the metal. A. B. MANNING.

Arc welding in hydrogen and other gases. P. ALEXANDER (Gen. Electric Rev., 1926, 29, 169—174; Chem. Zentr., 1926, I., 2886; cf. preceding abstracts).—In the production of atomic hydrogen for welding, the stream of pure hydrogen may be replaced by mixtures of hydrogen with nitrogen, carbon monoxide, or ammonia. Organic compounds, such as methyl alcohol, may also be used for the purpose. A. B. MANNING.

Influence of cathodic hydrogen on the strength of steel. D. ALEXEJEV and M. POLUKAROV (Z. Elektrochem., 1926, 32, 248—252).—Cathodic polarisation of a steel wire in pure sodium hydroxide solution leaves its strength entirely unaffected, but if a small quantity of mercury ions be present the steel may become as much as 20 times more brittle. In $0{\cdot}1N$-sodium hydroxide solution the effect increases with the concentration of mercury, reaching a constant value in a solution containing 0·00125 mg. per 100 c.c. The optimum current density for $0{\cdot}1N$-sodium hydroxide and 0·0025 mg. of mercury per 100 c.c. is 0·2 amp. per sq. cm. Polished steel becomes more brittle than steel simply cleaned by washing in ether or benzene. The reduction in strength is less at higher temperatures. Variation in the concentration of sodium hydroxide has a considerable influence; in a solution containing 0·0025 mg. of mercury per 100 c.c., at a current density of 0·2 amp. per sq. cm., and temperature 10°, there is a very pronounced maximum of brittleness when the strength of sodium hydroxide is $0{\cdot}25N$. In a solution of pure sulphuric acid the strength of the steel remains unchanged and mercury has no effect. The presence of small quantities of arsenic, however, produces a marked brittleness. The effect diminishes with increase of temperature, disappearing completely at 55—60° in $0{\cdot}1N$-sulphuric acid containing 0·025 mg. of arsenic per 100 c.c. with a current density of 0·08 amp. per sq. cm. The arsenic may be made ineffective, however, by rubbing the wire with a brush of glass wool during the electrolysis. The phenomenon is probably very complicated, but appears to depend on the formation of hydrogen as a secondary product of the decomposition of sodium amalgam or of arsenic hydride, not on the primary electrolytic formation. Hydrogen has been observed to penetrate steel, but an X-ray examination of the spacings between the atoms of iron suggests that the penetration of the iron by arsenic or sodium would be much more difficult. M. S. BURR.

Surface films in cathodic polarisation of metals. LIEBREICH.—See X.

PATENTS.

Protecting electric transformer oils against oxidation. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of COMP. FRANÇ. POUR L'EXPLOIT. DES PROC. THOMSON-HOUSTON (E.P. 212,928, 13.3.24. Conv., 13.3.23).—Oils used in electric transformers for insulating and cooling purposes are protected against oxidation by means of quinol suspended in the oil at or near the surface exposed to the oxidising influence. If desired the quinol may be placed in a porous container fitted into the upper part of the transformer or may be used in the form of an emulsion, either alone or mixed with a substance, *e.g.*, paraffin wax, which is fusible below the working temperature of the transformer and is soluble in the oil. J. S. G. THOMAS.

Conservation of transformer oils. ALLGEM. ELEKTRIZITÄTS-GES. (G.P. 426,996, 13.4.23).—While the transformer is running, basic or amphoteric oxides, carbonates, or silicates or refining earths, such as fuller's earth, are introduced into the transformer or into a separate device inserted in the oil-circulating system. Acid substances resulting from changes undergone by the oil are thus rendered harmless. R. A. A. TAYLOR.

[Depolariser for] dry batteries or batteries with a solidified electrolyte. SOC. ANON. "LE CARBONE" (E.P. 228,872, 20.1.25. Conv., 7.2.24).—A depolarising electrolyte for dry batteries consists of a coagulum of fecula containing powdered wood charcoal in suspension and an aqueous solution of ammonium chloride. J. S. G. THOMAS.

Electrolytes for secondary batteries. L. F. DUCKER (E.P. 250,448, 24.7.25).—An electrolyte for use in batteries having lead plates consists of a solution containing 1 lb. of a mixture of magnesium

sulphate (53%) and sodium sulphate (47%), 1 quart of commercial sulphuric acid (*d* about 1·835), in 3 quarts of water. J. S. G. Thomas.

Double-fluid [electric] cells. L. Darimont (E.P. 250,803, 30.6.25. Addn. to 241,729).—In a double-fluid cell of the type described in the previous patent, the layer of cement covering the wall of the porous vessel is coated with a pellicle formed of a solution of an alkali oleate or soap, which, reacting with suitable non-alkaline salts, *e.g.*, the ferric chloride of the depolarising solution and/or the zinc chloride of the exciting solution, produces a layer of insoluble fatty compounds forming a semi-permeable zone, which may be reinforced by addition of sodium phosphate, sodium carbonate, etc. to the alkali oleate employed. If desired, albuminous substances, *e.g.*, corn flour, gelatin, etc., may be added to the coating solution.
J. S. G. Thomas.

Apparatus for continuously cleaning insulators used in electrical gas cleaning and dust-precipitating installations. Lodge-Cottrell, Ltd. From Metallbank & Metallurgische Ges. A.-G. (E.P. 250,499, 17.12.25).—The electrical precipitator is provided with a sloping roof forming a passage with diminishing cross-sectional area, conveying the purified gases to a vertical outlet conduit. The bearer supporting the electrodes passes through openings in the walls of the conduit, and rests upon insulators supported by the sloping roof. A chamber enclosing the insulators, built around the sloping roof and the lower part of the conduit, is provided with openings at the top and bottom so that the heating effect of the sloping roof causes a constant current of hot air to pass upwards through the chamber, thereby keeping the surface of the insulators clean and free from moisture.
L. A. Coles.

Electron discharge devices. Standard Telephones and Cables, Ltd. From Bell Telephone Laboratories, Inc. (E.P. 250,485, 10.11.25).—An electron discharge device comprises a cathode of thermionically active material and a grid produced by intimately introducing into the material of which the grid is mainly composed a material, *e.g.*, aluminium or an aluminium compound, capable of conbining with the particles or vapour emitted from the cathode to form a thermionically inactive compound so that gases evolved within the device are fixed. Thus the grid may be formed of an aluminium alloy. The composite grid is subjected to heat treatment whereby the added material is either diffused through the substance of the grid or forms a homogeneous coating. J. S. G. Thomas.

Cold exhaustion of incandescence electric lamps and the like. D. S. Gustin, Assr. to Westinghouse Lamp Co. (U.S.P. 1,581,731, 20.4.26. Appl., 25.9.23).—Bulbs containing a filament to which a "getter" consisting of a mixture of phosphorus and graphite has been applied, are exhausted cold, flushed with oxygen, and again exhausted cold, after which the filament is flashed to remove the last traces of gas and moisture. L. A. Coles.

High-frequency apparatus and method of heating. I. Langmuir, Assr. to General Electric Co. (U.S.P. 1,579,009, 30.3.26. Appl., 23.5.24).—When a body to be heated by induction in a high-frequency field comprises a closed conductor such as a cylinder, a much more rapid heating is effected if the body is rendered discontinuous in the direction in which currents are induced. This result can be applied to heating liquids and gases by enclosing in a tube of suitable non-conducting material, such as glass, a number of coaxial discontinuous metallic cylinders which are heated by the action of an alternating field with a frequency above 10,000 cycles per sec. The gas or liquid to be heated is passed through the glass tube. T. S. Wheeler.

Process of making plates for storage batteries using lead suboxide. G. Shimadzu (U.S.P. 1,584,479, 11.5.26. Appl., 20.2.23).—See E.P. 198,647; B., 1924, 524.

See also pages 523, **Fuel drying plant with electrical precipitator** (G.P. 425,499). 524, **Electric flue-gas testing apparatus** (E.P. 250,478). 525, **Separating emulsions** (U.S.P. 1,570,209); **Electrical dehydrator** (U.S.P. 1,579,515); **Cracking tars and oils** (E.P. 248,830). 526, **Cracking hydrocarbon oils** (U.S.P. 1,579,554 and 1,579,601). 560, **Refining sugar juice by chlorination** (E.P. 249,191). 561, **Decomposing barium saccharate** (G.P. 425,170).

XII.—FATS; OILS; WAXES.

China wood [tung] oil. K. H. Bauer (Chem. Umschau, 1926, **33**, 53—56).—Pure α- and β-elæostearic acids were heated to 200° in an atmosphere of carbon dioxide for varying lengths of time, the volatile decomposition products being collected and the changes followed by determination of the chemical constants, by hydrogenation of the products, and by molecular weight determinations. Probably anhydride formation from the free α- or β-acid, together with the formation of some other saponifiable compound, occur during the heating, and examination of the volatile products evolved on heating the β-acid indicates that "cracking" may have occurred. The polymerised β-acid may be a mixture of different acids partially of higher and partially of lower molecular weight than elæostearic acid, with different numbers of unsaturated groups. Molecular weights as determined in camphor solution are in all cases about half the corresponding values found in benzene solution, but these determinations give no definite indications of the changes which have occurred. The author contrasts the results obtained with those obtained by Nagel and Grüss (B., 1926, 164) on heating methyl α- and β-elæostearates at different temperatures in carbon dioxide. H. M. Langton.

Action of sulphur on linseed oil. G. S. Whitby and H. D. Chataway (J.S.C.I., 1926, **45**, 115—121 T).—To samples of 20 g. of raw linseed oil definite

amounts of sulphur up to 16·5% were added with agitation and exclusion of air, and at temperatures maintained accurately at 160° (or 170° in some cases), samples being withdrawn at intervals prior to setting and the amount of acetone-insoluble fraction determined. In all cases after heating for 45 min. at 160° all the sulphur added had combined with the oil. Results are given for time of setting and for the concentration of acetone-insoluble material at the moment of gelation. The proportion of sulphur in combination with the oil was always higher in an acetone-insoluble than in a corresponding acetone-soluble fraction. In the action of sulphur and heat on drying oils three well-defined stages succeed each other, viz., chemical combination of sulphur with the oil which takes place comparatively quickly, followed by the formation of an acetone-insoluble product in the second stage, a gradually increasing proportion of the oil changing to a more viscous liquid insoluble in cold acetone. This change is apparently polymerisation, the speed of which is greater, the greater the amount of sulphur present and the higher the temperature. Finally, gelation takes place, and it is presumed that during heating the acetone-insoluble material gradually assumes colloidal dimensions and this colloidal material imbibes the acetone-soluble liquid and any portion of the acetone-insoluble liquid still non-colloidal and a gel then appears rather suddenly. The closely related phenomena of the "drying" of oils and of their thickening under the influence of heat are shown to be similar to the setting of oils under the influence of sulphur and heat. H. M. LANGTON.

Effect of "blowing" on the composition of certain fatty oils. C. H. THOMSON (Analyst, 1926, **51**, 177—180).—The effect of "blowing" at different stages has been examined in the case of cotton seed, whale, sperm, and shark oils, the oils being treated on the large scale, in quantities of about 4 tons, at 115°. Samples were taken at different stages and the specific gravity, viscosity at 93° (Redwood), and the usual constants and variables were determined. Methylated ether was the solvent used to extract the unsaponifiable matter. Tabulated results indicate that iodine value and unsaponifiable matter fall during the oxidation, while specific gravity, viscosity, saponification value, refractive index, and free fatty acids (as oleic) rise to a greater or less extent. Although the constants and variables rise or fall simultaneously at each stage they do not do so at the same rate. The rise in saponification value is concurrent with the rise in soluble volatile acids as indicated by the Reichert–Wollny value. The rise in acid value is also concurrent with the fall in unsaponifiable matter, the increase in acid value being apparently partly due to the oxidation of the unsaponifiable alcohols to fatty acids. Ether-insoluble bromides are not produced from blown shark, whale, or sperm oils, though they are given by the untreated oils. There is practically a regular rise in the viscosity and refractive index after the oil reaches a viscosity of 700 sec., and up to 1800 sec., but below 700 sec. the relationship is irregular. The various changes caused by "blowing" are brought out clearly in a table giving the analysis of the saponified oils. The amount of glycerol produced is not affected but a considerable quantity of apparently neutral organic matter is formed. Similarity in conditions of blowing must be observed to obtain similar results—the changes in composition are in the same direction as the blowing is continued. In the case of sperm oil blowing was also carried out with the addition of 0·1% of cobalt oxide and certain anomalies were noted. H. M. LANGTON.

Detection of coconut oil in cacao butter and chocolate. J. RUFFY (Ann. Chim. analyt., 1926, 8, 131—134).—Quantities of coconut oil between 1 and 30% may be detected in cacao butter by applying the method suggested by Härtel and Maranis (B., 1924, 571), but certain modifications were found necessary. The Reichert–Meissl value is determined after saponification with 4 g. of glycerin and 2 c.c. of 1:1 potassium hydroxide solution, the 110 c.c. of distillate being filtered into a 300 c.c. flask and boiled for 15 min. with an upright condenser before titrating. The titrated liquid is slightly acidified, made up to 200 c.c., and 100 c.c. are redistilled and titrated (B). If the value of 100 B/R-M value exceeds 60 adulteration of the cacao butter is to be suspected, 1—2% of coconut oil raising it to about this figure. For milk chocolate the index value must be over 80 before adulteration is assumed. D. G. HEWER.

Lime pip oil. S. C. MARSHALL and M. S. SALAMON (Analyst, 1926, **51**, 237—238).—A crude reddish-brown oil of bitter flavour obtained from West Indian lime pips had the following characteristics: $d^{15\cdot5}$ 0·925, n_D^{40} 1·465, free acidity as oleic acid, 2·8%, saponification value 193·5, iodine value (Wijs, 1 hr.) 109, unsaponifiable matter 0·8%, clouding point in Bellier's test 17·5°. The fatty acids showed n_D^{40} 1·4565, neutralisation value 198, titre 35·8°. The alkali-refined oil (pale yellow and free from bitterness) had $d^{15\cdot5}$ 0·921, n_D^{40} 1·4645, and clouding point in Bellier's test 18·4°. D. G. HEWER.

Chemical reactions in fat hardening. W. NORMANN (Z. Deuts. Oel- u. Fett-Ind., 1926, 46, 193—195).—Mainly a discussion of the general reactions involved and the behaviour of the nickel–kieselguhr catalyst. Graphs are given for a number of oils showing the progress and completion of hardening, the fall in iodine value and corresponding rise in m.p., and also the fall in iodine value together with progressive fall in refractive index (Zeiss butyrorefractometer). For judging the progress of hardening in a particular case the determination at intervals of the refractive index of the oil is the quickest and is better than a method involving the measurement of the amount of hydrogen consumed. An accompanying table shows the amount of hydrogen in cub. m. at 0° and at 20° required to saturate completely 1000 kg. of the commoner fatty acids and fatty oils. In practice oils are seldom hardened till completely saturated. Owing to loss of hydrogen and to side reactions the amount

of hydrogen consumed in a particular hardening is in practice 10—20% in excess of the amount as calculated from the fall in the iodine value.
H. M. Langton.

Determination of salt in soap. J. Davidsohn (Chem. Umschau, 1926, 33, 89—90).—Mainly a review of the various methods of determining common salt in soap with particular reference to the method due to Bennett (B., 1921, 741 A). The method gives useful results, but it is preferable to filter off the precipitate of magnesium soap before titrating.
H. M. Langton.

Influence of rate of stirring on reaction velocity. Huber and Reid.—See I.

Rancidity and oxidation of fatty oils. Rhys-Davies.—See V.

New value in examination of fat mixtures. Van Raalte.—See XIX.

Patents.

Conversion of neutral or nearly neutral triglycerides, mutually or with other esters. C. Van Loon (E.P. 249,916, 30.12.24).—Neutral or nearly neutral glycerides are heated for such a time that an interchange of the acid radicals takes place; substances are added which promote catalytically the esterification of free fatty acids and free alcohols, or the action of free alcohols or free acids on triglycerides. For example equal parts of neutral coconut oil and ethyl stearate with the addition of 2% of tin powder were stirred at a temperature of 250°. A slow current of carbon dioxide, to inhibit oxidation, was passed over the mixture and after 1½ hrs. this current was accelerated in order to distil off the more volatile ethyl esters of the coconut oil fatty acids formed during the reaction. A practically neutral distillate of saponif. value 245 (calc. for ethyl stearate 180, and for ethyl laurate 246) was obtained, indicating conversion of the ethyl stearate into ethyl esters of coconut oil fatty acids. In the absence of a catalyst no lower ethyl esters could be isolated even after prolonged heating. Other metals, in addition to tin, and compounds of metals and also organic sulphonic acids may be used as catalysts. H. M. Langton.

Manufacture of a detergent. C. W. Fulton and H. W. Hutton (E.P. 249,912, 6.12.24).—At least two oils, fatty acids, or resins ordinarily saponifiable are sulphonated. Unsulphonated soap-making oils are then emulsified with the acidulated oils and caustic alkali is added to saponify the soap-making oils and neutralise the acidulated oils. For example coconut oil (1½ pts.) is sulphonated with 25% of its weight of sulphuric acid of *d* 1·84, and castor oil (1 pt.) is separately sulphonated with 25% of its weight of sulphuric acid. The acidulated oils washed with salt or fresh water, are emulsified together with 3 pts. of unsulphonated soap-making oil, such as cottonseed oil. The whole is warmed and treated with sufficient caustic soda to produce a compound sulphonated soap stock. H. M. Langton.

Soaps and detersive preparations and manufacture thereof. R. Vidal (E.P. 250,108, 30.9.25. Addn. to 228,390).—Soaps and similar preparations obtained as described in the chief patent (B., 1925, 251) are mixed with aqueous solutions of alkali hypochlorites. For example the pasty soap from 60 pts. by wt. of an aqueous solution containing 33% of castor oil in alkali combination, and 20 pts. of free fatty acid (oleic acid, copra acids, etc.) is added gradually to 80 pts. by wt. of an aqueous solution of sodium hypochlorite at 10°B. (*d* 1·07). The resulting preparations are easily soluble in water to a clear solution.
H. M. Langton.

Combining gases and vapours with liquids. [Hydrogenation of oils.] S. Ruben (E.P. 251,134, 16.6.25).—See U.S.P. 1,554,296; B., 1925, 999.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Influence of the hardening period upon the protective influence of paints. J. N. Friend and B. L. Griffin (J. Oil and Colour Chem. Assoc., 1926, 9, 106—108).—During experimental work on a standard paint for use as a criterion for rust-preventive coatings, a series of mild steel plates were coated with a paint consisting of 80 pts. of Turkey-red oxide of iron and 100 pts. of raw linseed oil (no thinners or driers), and exposed to corrosion by immersion in rain water. The loss in weight of the plate after removal of paint and rust was found to diminish markedly as the interval between painting and exposure was prolonged, the corrosion being three and a half times greater when the time of hardening was 11 days than when the film was allowed to harden for 300 days before exposure. For one coat of this particular paint, a hardening period of about 250—300 days seemed to be a limiting value beyond which no additional protection was afforded. S. S. Woolf.

Water-line paints [ships' "boot-topping"]. M. Ragg (Farben-Ztg., 1926, 31, 1795—1798).—The results obtained by Figg in an exposure test of water-line paints (J. Oil and Colour Chem. Assoc., 1925, 8, 314—334) are described and criticism is offered mainly on the ground that the panels were unavoidably left to harden for 65 days before exposure. This is divorced from practical ship-painting conditions, as a vessel has to be repainted etc. in the interval between voyages—a matter of a few days generally. It is also affirmed that the weathering effects on stationary iron structures subjected to tides are much less drastic than those on the water-line zone of sea-going ships. Some of the experimental paints prepared by Figg were too rich in volatile thinner, and all were too slow drying.
S. S. Woolf.

Lead resinates. C. Coffignier (Rev. gén. Coll., 1926, 4, 1—8, 65—74).—10, 15, 20, and 25% solutions in benzene, turpentine oil, and white spirit (light petroleum) of resinates of lead containing 5 and 10 pts. litharge to 100 pts. of rosin respectively were examined over a period of two months for

viscosity changes, amount and nature of sediment etc. (in the case of the 5% resinate in white spirit observations up to 6 months are recorded). The relative stabilities of the solutions are discussed (cf. B., 1925, 815). S. S. WOOLF.

Copal melting and the copal-melting process in closed kettles. H. RAHDER (Farben-Ztg., 1926, **31**, 1742—1743).—Gum-running is a process of the utmost importance in varnish manufacture, but although the presence of air is known to be detrimental, open pots are largely used to-day throughout the industry. The preparation of varnishes in a closed glass retort showed that the removal of air and introduction of inert gases such as carbon dioxide give rise to a lighter product than can be prepared in an open vessel, at the same time permitting the use of higher temperatures than are customary as well as reducing fire risks and assisting the even heating of the mass. The experiments have been extended to the technical scale, and by means of various inlet tubes, the inert gas may be introduced at different places in the kettle, to suit the particular process occurring. Since greater latitude in temperature becomes possible owing to the exclusion of air, control is achieved by fixing temperature and time, without resort to the withdrawal of samples at intervals to ascertain progress. The closed kettle can also be used for oil-thickening and the preparation of glyceryl resinates (ester gums), but heating under reduced pressure presents new, and at present unsurmounted, difficulties. S. S. WOOLF.

Examination of sandarac. H. WOLFF (Farben-Ztg., 1926, **31**, 1682—1683).—Eighteen samples of African (Mogador) sandarac and two samples from Asia Minor showed considerable divergence amongst themselves, when examined for acid value, saponif. value, and percentage soluble in light petroleum, the last (about 15% for the African and about 25% for the Asia Minor samples) being found to be higher than previously reported in the literature. In view of the high acid value of sandarac and the fact that the light petroleum extract gives an orange-red coloration with the Liebermann–Storch reagent, the presence of rosin as an adulterant should not be regarded as proven unless gelatinisation occurs when a benzol solution of the light-petroleum extract is treated with ammonia, in addition to the production of a violet-red coloration in the Liebermann–Storch test. S. S. WOOLF.

Analysis of oil varnishes. A. GÉRARD-VAUDIN (Ann. Falsif., 1926, **19**, 267—275).—A review of published methods.

China wood oil. BAUER.—See XII.

Action of sulphur on linseed oil. WHITBY and CHATAWAY.—See XII.

Effect of blowing on composition of oils. THOMSON.—See XII.

PATENTS.

Compositions containing synthetic resins. G. PETROFF (E.P. 231,431, 16.1.25. Conv., 25.3.24).—The liquid condensation products obtained by warming phenol with formaldehyde in the absence of a catalyst are mixed with hydrocellulose or oxycellulose, alone or together with acid catalysts and with materials such as disintegrated infusible phenol—aldehyde condensation products, barium or calcium sulphate, or kaolin, moulded into shape at the ordinary temperature, and then heated to expel water and effect the final condensation. For example, the oily condensation product from 100 pts. of phenol and 110 pts. of 40% formaldehyde, after removal of the aqueous layer, is mixed with 2 pts. of benzenesulphonic acid, 5 pts. of 40% formaldehyde, and 110 pts. of hydrocellulose. When the mixture has attained a suitable consistency and strength, it is moulded into shape, and dried and hardened by heating gradually to 110°. L. A. COLES.

Varnish and paint remover. M. PHILLIPS and M. J. GOSS ; dedicated to the People of the United States (U.S.P. 1,580,914, 13.4.6. Appl., 24.10.25).—A mixture of *p*-cymene and 95% ethyl alcohol is claimed. S. S. WOOLF.

Paint. G. SHIMADZU (U.S.P. 1,584,152, 11.5.26. Appl., 18.4.24).—See E.P. 215,222 ; B., 1924, 566.

Corrosion apparatus [for manufacture of white lead]. C. P. TOLMAN, Assr. to NATIONAL LEAD CO. (U.S.P. 1,584,906, 18.5.26. Appl., 26.1.22).—See E.P. 186,114 ; B., 1922, 905 A.

Cracking tars and oils (E.P. 248,830).—See II.

Lignone derivatives (E.P. 248,834).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Bulk test with *p*-nitrophenol [as mould-preventive for rubber]. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1926, **8**, 177).—Large samples of smoked sheet and pale crêpe rubber prepared from latex by coagulation with acetic acid containing *p*-nitrophenol in solution were mould-free, of fairly good appearance, and of normal vulcanisation behaviour. D. F. TWISS.

Chemical analysis of rubber. C. J. ENKLAAR (Chem. Weekblad, 1926, **23**, 209—210).—Different rubbers may be characterised by means of the fractions obtained by dry distillation. A sample of Java crêpe so treated yielded nearly 3% of isoprene; a sample of sheet yielded no isoprene, but products resembling dimethylbutadiene were obtained from it. S. I. LEVY.

Colloid-chemical processes during hot vulcanisation. H. POHLE (Kolloid-Z., 1926, **39**, 1—7).—The nature and degree of dispersion of sulphur in mixtures of rubber and magnesium carbonate, lime, litharge, golden antimony sulphide, and the structure of Congo rubber were examined with the aid of the microscope, and in some cases, the ultra-microscope,

some of the specimens being unvulcanised and others vulcanised. The analogy between the effect of vulcanisation and that of reinforcement by amorphous carbon is again indicated; the probability that vulcanisation involves the formation of an additional disperse phase is confirmed by the concomitant increase in the Tyndall effect. No theory of vulcanisation which disregards the heterogeneous disperse character of the reaction mixture can be satisfactory.
D. F. Twiss.

Effect on mechanical properties of the formation of colloidal precipitates during vulcanisation of rubber. G. Martin and W. S. Davey (J.S.C.I., 1926, **45**, 174—176 T).—Mixings containing acetone-extracted rubber, zinc oxide, and sulphur do not yield more than a trace of zinc sulphide on vulcanisation. In the presence of fatty acids or artificial accelerators appreciable quantities of zinc sulphide are formed. The zinc sulphide appears to be very finely divided but, contrary to Bysow's theory of vulcanisation, it has no vulcanising effect. Moreover, it has no appreciable effect on the physical properties of the vulcanised product.

Vulcanisation of "diffusion-caoutchouc." H. Feuchter (Gummi-Ztg., 1926, **40**, 1691—1692; cf. B., 1925, 557).—"Diffusion-caoutchouc," *i.e.*, the fraction extractable from deresinised smoked sheet rubber by light petroleum at the ordinary temperature, can be vulcanised in the normal manner when mixed with sulphur, zinc oxide, and piperidine piperidinecarbothionolate. The vulcanised product, however, ages badly. D. F. Twiss.

Variations in the tensile strength of rubber-sulphur vulcanisates. B. J. Eaton and R. O. Bishop (Malay. Agric. J., 1926, **14**, 53—64).—Contrary to the results of earlier workers, the authors find that the addition of mineral particles from the soil does not decrease the tensile strength nor cause any definite increase in the deviation from the mean breaking load; the addition of bark reduces the average tensile strength. The variations in the tensile strength of plantation rubber are not entirely fortuitous, but do not depend on the presence of "dirt."
D. F. Twiss.

Physical properties of rubber. I. Influence of high temperatures on the tensile curve of vulcanised rubber. A. van Rossem and H. van der Meyden (Kolloid-Z., 1926, **39**, 69—80; Proc. K. Akad. Wetensch. Amsterdam, 1926, **29**, 296—305).—See B., 1926, 502.

Patents.

Jelutong product. Beech-Nut Packing Co., Assees. of W. A. Beatty (E.P. 245,405, 13.1.25. Conv., 30.12.24).—Jelutong or pontianac produced by evaporation of fresh or preserved latex contains 20—25% of benzene-insoluble solids. These involve a corresponding increase in the yield and effectively protect the product against atmospheric oxidation, whereas the product obtained by coagulation undergoes rapid oxidation when dry. The evaporated product also has a pleasant taste and odour, and can be used, without any refining process, for the manufacture of chewing gum. D. F. Twiss.

Manufacture of rubber compositions. W. B. Wiegand (E.P. 250,279, 29.9.24).—A high percentage of finely-divided powder, *e.g.*, carbon black (50% and upwards on the rubber content), can be incorporated into natural or concentrated latex by previously adding to the latter an anti-coagulant such as ammonia and an organic substance or substances, *e.g.*, gelatin, capable of lowering the surface tension or increasing the stability of the latex; the powder is introduced as an aqueous emulsion or paste, which also may contain ammonia and a suitable organic material, *e.g.*, saponin. The final addition of a little oleic acid to the mixture increases the degree of dispersion and the stability. Other ingredients such as vulcanising agents and colours may be introduced, together with the emulsified powders. (Reference is directed in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 214,210, 212,597, and 205,487.) D. F. Twiss.

Production of combinations of rubber and paper. E. Hopkinson and R. P. Rose, Assrs. to Gen. Rubber Co. (U.S.P. 1,567,646, 29.12.25. Appl., 22.10.25).—In a mixture of paper pulp and rubber latex the rubber is agglomerated, and then, after further mixing, coagulated. D. F. Twiss.

Method of making moulded rubber articles, mould and lubricant therefor. B. Dales and W. S. Cable, Assrs. to B. F. Goodrich Co. (U.S.P. [A] 1,580,966 and [B] 1,580,967, 13.4.26. Appl., 17.11.24 and 20.11.24).—The mould surfaces which make contact with the rubber during vulcanisation are coated, at least in part, with a substantially water-insoluble composition comprising (A) a wax resistant to absorption by rubber at temperatures of vulcanisation, or (B) sugar-cane wax. D. F. Twiss.

Drying coagulable liquids [rubber latex]. E. Hopkinson and R. P. Rose, Assrs. to Gen. Rubber Co. (U.S.P. 1,582,604, 27.4.26. Appl., 18.7.23).—A layer of latex is evaporated on a surface from which the dry residue is continuously removed.
D. F. Twiss.

Production of a vulcanising accelerator for rubber. C. O. North (E.P. 251,005, 20.1.25).—See U.S.P. 1,467,984; B., 1923, 1083 A.

XV.—LEATHER; GLUE.

Finer structure of the collagen fibre. A. Küntzel (Collegium, 1926, 176—189; cf. B., 1925, 966).—The collagen fibre consists of fine fibrils of indefinite length, 1×10^{-4} cm. in diameter. The elastic fibres consist of a basic collagen material sheathed by a substance which gives the fibres their characteristic properties. Pancreatin destroys the encasing material but not the whole fibre. The former gives the characteristic colour reactions of elastin, and these show that the gradual decomposition of this substance begins in the middle of the fibrils. Apparently the core of the fibril is the least

resistant and the outer portion the most resistant to reaction. The fibrils are built up of concentric layers. It is probable that the collagen fibril is similarly constituted since the fine fibrils of the grain are more difficult to lime than the coarser fibrils. The finer fibrils are merely outer layers, hence their greater resistance. The fibrils consist of chains of micelles arranged in the direction of the fibre and united by cementing substance, according to the Nägeli theory which explains swelling and taking up of water. The micellar theory explains the double refraction observed with collagen fibrils and the reversal of the double refraction by tanning materials. Gelatin micelles are not the same as collagen micelles.

D. Woodroffe.

Industrial determination of tannin. L. Meunier and A. Jamet (Chim. et Ind., 1926, **15**, 499—505).—A critical résumé of the methods used for determining tannin. The official "shake method" is essentially empirical and requires a rigorous standardisation of the conditions under which it is applied, particularly with regard to the clarification and concentration of the solution, the preparation of the hide powder, and the proportion of the latter used. Centrifuging the solution is preferable to filtration through a Berkefeld filter, which removes part of the tannin present in colloidal form. The official method has the advantage of reproducing more nearly the conditions under which the tannin solutions are actually used, but the hide powder adsorbs material other than tannins from the solution. The method of Wilson and Kern (B., 1921, 819 A) gives useful information on the amount of material reversibly and irreversibly adsorbed respectively.

A. B. Manning.

Concentration factor in the fixation of chromium compounds by hide substance from chromium chloride solutions. K. H. Gustavson and P. J. Widen (Collegium, 1926, 153—165).—Experiments with chromium chloride liquors of 12·8%, 33·0%, and 54·6% basicity and different p_H values and concentration show that the amount of chromium fixed increases and the p_H of the liquors decreases with increased concentration. The p_H of chromium chloride liquors increases after dilution and keeping for 48 hrs., which contrasts with the behaviour of chromium sulphate liquors on dilution. The greatest increase in p_H was observed on keeping very basic chromium chloride solutions containing 20—40 g./litre of chromium. The p_H increases in solutions weaker than 10 g./litre, decreases in stronger solutions, and then increases again at greater concentrations although the liquors have a greater actual acidity in the more concentrated solutions. The different behaviour of chromium sulphates and chlorides is intimately connected with the marked difference in the tendencies of sulphate and chloride groups to form complexes and with the changes which result in the inner sphere. The function of the hydroxyl groups in the complex cation influences the activity of the liquors after dilution. The p_H value of used liquors diminishes on dilution. The p_H curves show a steep rise with diminishing basicity figure. The amounts of chromium and chlorine fixed by the hide substance increase with increase in concentration. In all cases the basicity of the chrome-collagen compound is much higher than that of the original liquor, a difference which is more marked with acid liquors. This is very different to the results obtained with sulphate liquors. The amount of chromium fixed by hide substance increases with increase in the basicity of the liquors. The hydrogen-ion concentration does not affect the tanning power very much.

D. Woodroffe.

Changes in the "true tanning figure" during tannage. G. Powarnin and J. Esrochi (Collegium 1926, 165—176).—Pieces of dried calfskin were tanned with quebracho, mimosa, and willow bark respectively, first in weak liquors, gradually strengthening each day, and finally in a drum with stronger liquors. In one series the pieces of pelt were swollen with ½% sulphuric acid solution before tanning. The preliminary treatment with sulphuric acid solution diminished the tensile strength of the leather but increased the true tanning figure (cf. B., 1923, 1141 A) by 10—16%. The moisture content of the leather and its tensile strength were connected. The true tanning figure was found to increase during the first two weeks of pit tanning, then diminish slightly during the third week, still more during the fourth week, show a large increase after the drumming in the first liquor, but another decrease after drumming in a stronger liquor. The sum of the "true tanning figure" and the "weighting figure" showed a continuous increase during the tannage, 43·85% after 1 week in quebracho liquors to 112% after 4 weeks in liquors and drumming for 24 hrs. This sum was 156% for leather previously swollen with weak acid, compared with 125% for leather tanned unswollen. The tensile strength depends on the moisture content of the leather and also on the "true tanning figure."

D. Woodroffe.

Properties of shoe leather. III. Ventilating properties. J. A. Wilson and R. O. Guettler (J. Amer. Leather Chem. Assoc., 1926, 21, 241—250).—The porosity of leather and its permeability to water vapour, described as ventilating properties, have been measured for the 18 different types of shoe leather described previously (B., 1926, 504) by determining the rate of passage of water through leather from an atmosphere of high to one of low relative humidity. A large amount of water passed through leather as compared with that passing through free space and it increased in the same way with rise of temperature. It was directly dependent on the difference in relative humidity between the two spaces and was constant at 66% of that passing through free space at all relative humidities. The porosity to air and permeability to water vapour both diminished as the amount of grease or oil in the leather was increased, but they bore no direct relation to each other. The application of casein to the grain of the leather diminished the porosity but had little effect on the permeability. The application of collodion diminished both. Chrome

sole and patent leathers had a very small permeability to water vapour and no porosity; suede had maximum ventilating properties. Vegetable-tanned sole leather had a fair degree of permeability but low porosity. D. Woodroffe.

Variation in tensile strength of calf leathers with humidity. J. A. Wilson and E. J. Kern (J. Amer. Leather Chem. Assoc., 1926, 21, 250—257). Tensile strength tests on chrome- and vegetable-tanned calfskin pieces at different relative humidities have shown that the strength of chrome leather increases considerably (50%) with increased relative humidity, whereas that of vegetable-tanned leather increases only slightly (13%). The greater strength at higher relative humidities is probably due to the lubricating value of the absorbed water. The greatest amount of water absorbed by the vegetable-tanned leather was 30·18%, against 70·37% for the chrome leather. D. Woodroffe.

Bone glues. J. C. Kernot and (Miss) N. E. Speer (Second Report, Adhesives Research Committee, 1926, 17—22).—Maceration of bones in dilute alkali before extraction improves the quality of the glue. A short period of treatment under high pressure is preferable to a long period under low pressure for the extraction of undecalcified degreased bones. Air pressure gives the best results; if steam is used, this should be as dry as possible, as wet steam will cause hydrolysis of the gelatin and yield a glue of poor quality. B. W. Clarke.

Production of glue and gelatin from fish. J. C. Kernot and (Miss) N. E. Speer (Second Report, Adhesives Research Committee, 1926, 23—33).—Fish skins, *e.g.*, the waste from filleting factories, can be used for the manufacture of odourless glue of good quality if subjected to preliminary maceration with dilute alkali and dilute acid (cf. E.P. 235,635; B., 1925, 683). The glue produced compares fairly well with the best hide or bone glue, and has a greater resistance to moisture. The process must be operated in conjunction with the manufacture of fish meal to be commercially successful. B. W. Clarke.

Gelatin. S. B. Schryver (Second Report, Adhesives Research Committee, 1926, 7—16).—Gelatin may be purified by electrolysis and subsequent crystallisation, but the purest form is obtained by flocculation of a dilute solution in an electric field. A method has been developed for the analysis of proteins by precipitating the amino-acids as carbamates, by which at least 18 amino-acids can be separated. The Hausmann number is not satisfactory, since it is affected by the pre-treatment of the gelatin, and the treatment of the precursor must be taken into account (cf. Knaggs, Manning, and Schryver, B., 1923, 1084 A; Knaggs, B., 1924, 955; Kingston and Schryver, B., 1924, 955; Knaggs and Schryver, B., 1924, 955, 956; Manning, B., 1924, 955; Horne, B., 1924, 956; Schryver, Buston, and Mukherjee, B., 1925, 521). B. W. Clarke.

Adhesives and adhesive action. J. W. McBain and D. G. Hopkins (Second Report, Adhesives Research Committee, 1926, 34—89; cf. B., 1925, 292; 1926, 291).—An adhesive joint may be mechanical, due to the embedding of a tenacious film of adhesive in a porous surface, or specific, between two smooth non-porous surfaces. Metal joints capable of withstanding a pull of $\frac{3}{4}$ ton per sq. in. have been set up with gum arabic, and up to 3 tons with shellac–creosote cements. The highest results are obtained with the thinnest films provided complete contact is obtained with the surfaces. The most accurate results are obtained by measuring the strength of thin films of adhesive, which is often much greater than that of joints set up with the adhesive in the usual way. Gelatin adhesives are the best for wood; high-grade gelatins and glues give practically the same values (12,000 lb. per sq. in. tensile strength). The adhesive properties of gelatin are only slightly affected by prolonged heating at 60°, and of glue at 80°, but higher temperatures produce more marked deterioration. The addition of fillers produces little effect on the strength of the joint. The strongest sodium silicate joints are obtained with the neutral silicate $Na_2O,3SiO_2$. B. W. Clarke.

Mechanical tests of adhesives for timber. Royal Aircraft Establishment (Second Report, Adhesives Research Committee, 1926, 90—121).—The double cover plate type of joint gives less variable results for the strength of glued joints than either the simple lap or cantilever type. The energy absorption type is being developed, and shows promise of being a more accurate method. In this method the energy necessary to pull off a given area of cotton webbing which has been glued to a piece of walnut-wood is measured. The variations obtained by present methods render the interpretation of results very difficult. B. W. Clarke.

Examination of glued joints by *X*-rays. (Second Report, Adhesives Research Committee, 1926, 122—123).—*X*-Ray examination indicates the presence of irregularities in glue films provided a suitable filling agent, *e.g.*, lead sulphate, is added to the glue. The method promises to be useful in the detection of defective glued joints in wood structures. B. W. Clarke.

Patents.

Products useful in tanning and tawing. M. Kahn, E. Le Breton, and G. Schaeffer (E.P. 247,977, 20.11.24. Conv., 20.11.23).—Brewers' or distillers' yeast, *Rhizopus*, *Amylomyces*, or the like is autolysed, and the autolysate is separated. The cellulosic residues heated to 50° or 80°, and with or without addition of antiseptics, are mixed with fats, oils, soaps, or the like, which may or may not be heated to 40—80° before mixing. Fish may be mixed with yeast, the skeleton of the fish and the autolysate separated from the undigested portions, and an emulsion of fish oil in cellulosic residues so obtained. The products may be used in tanning and tawing. D. Woodroffe.

Tanning substances. Farw. vorm. Meister, Lucius, & Brüning, G. Kränzlein, A. Voss, and H. Gärtner (E.P. 250,398, 24.4.25. Addn. to 211,145 and 240,003; B., 1925, 605, 933).—A phenolic resin is combined with an aromatic or aliphatic compound containing one or more sulphonic groups capable of being exchanged easily, or a phenolic compound is condensed with an aromatic or aliphatic compound containing one or more sulphonic groups capable of being exchanged easily. The product in the latter case may be heated or treated with an agent capable of transforming phenols into phenolic resins. The products are tanning agents.
D. Woodroffe.

Treatment of hides, skins, etc. C. J. M. M. Le Petit, Assr. to J. S. Burns (U.S.P. 1,585,395, 18.5.26. Appl., 11.12.23).—See E.P. 216,546; B., 1924, 644.

Soaking liquor for dried hides and the like. D. J. Lloyd and R. H. Pickard, Assrs. to Brit. Leather Manufacturers' Research Assoc. (U.S.P. 1,584,462, 11.5.26. Appl., 20.5.24).—See E.P. 221,058; B., 1924, 919.

XVI.—AGRICULTURE.

Determination of the p_H of soils by means of Biilmann's quinhydrone electrode. D. J. Hissink and J. van der Spek (Verhdl. zweiten Komm. int. bodenk. Ges., 1926, Sept., 12 pp.; Chem. Zentr., 1926, I., 3099).—In using this method, the final reading can be made after 6—10 min. The soil suspension should be as concentrated as possible, and it is better not to add potassium chloride.
C. T. Gimingham.

Titration curves for humic soils. D. J. Hissink (Verhdl. zweiten Komm. int. bodenk. Ges., 1926, Sept., 20 pp.; Chem. Zentr., 1926, I., 3099).—In the determination of the "lime-requirement" of soils by means of potentiometric titrations, equilibrium is reached in about 3 days. Titration with calcium hydroxide gives more satisfactory results than with sodium hydroxide. Titration should be to the neutral point, not to equivalence.
C. T. Gimingham.

Can the "lime-requirement" of a soil be deduced from the reaction of a suspension of the soil in potassium chloride solution? M. Trénel (Mitt. Int. Bodenk. Ges., 1925, 1, 215—221; Chem. Zentr., 1926, I., 2831).—A continuation of earlier work (cf. B., 1925, 328). Since, in "exchange acid" soils, the p_H is a function of the "exchangeable" aluminium ions, the relation between "lime-requirement" and hydrogen-ion concentration can be expressed graphically. The curves for humic soils are clearly distinguished from those for mineral soils, and indicate a higher "lime-requirement" for the latter.
C. T. Gimingham.

Recognition of capacity of soil for nitrogen fixation. S. Winogradsky (Compt. rend., 1926, 182, 1061—1063).—The process of nitrogen fixation in the soil depends on the density of *Azotobacter*, this being in its turn dependent on the fertility of the soil towards the bacteria. In a preliminary test a silica-gel plate 9 cm. in diameter is inoculated with 50—100 grains of the soil, and maintained at 30° for 48 hrs. If the soil is active, 25—50% of the grains should develop colonies of *Azotobacter*; if slightly active, the colonies appear more slowly (4—5 days) and are fewer in number. Incorporation of 0·5 g. of mannitol with 50 g. of soil, followed by similar treatment, should result in spontaneous growth of *Azotobacter* if the soil is active. Similar treatment with 5% of starch should cause formation of colonies in considerable numbers. 1 g. of dry soil spread on a silica-gel plate of 20 cm. diameter, should yield 800—1000 colonies per 100 sq. cm. Determination of the nitrogen fixed by active soil in presence of mannitol should show 20 mg. fixed per 2 g. of mannitol in 120 hrs. With soils of low activity spontaneous growth of *Azotobacter* on addition of mannitol is not observed.
H. J. Evans.

Carbon dioxide and plant production. E. H. Reinau (Z. angew. Chem., 1926, 39, 495—503; cf. B., 1925, 142, 1002).—A general account of the part played by carbon dioxide in the production of farm and garden crops. The effects of various conditions on the percentage of carbon dioxide in the air, and on the amount evolved from the soil, and the results of the interactions between plant and soil on the distribution of carbon are discussed. The use of artificial means for increasing the carbon dioxide content of the air in the neighbourhood of growing plants is advocated, and the "Oco" process for this purpose, involving burning a special form of compressed carbon, is briefly described.
C. T. Gimingham.

See also A., May, 545, **Effect of metallic aluminium on nitrogen-fixing bacteria** (Truffaut and Bezssonoff); **Bacterial oxidation of sulphur** (Guittonneau). 547, **Rapid method for demonstrating effects of plants on a culture solution** (Hibbard).

Spent molasses wash as fertiliser. Cerasoli.—See XVIII.

Patents.

Apparatus for treating acid phosphate [superphosphate]. E. H. Armstrong (U.S.P. 1,576,022, 9.3.26. Appl., 2.12.22).—Phosphates and the like are passed through a tower, or casing, fitted with a series of conical guides spaced a suitable distance apart and with a rotating support carrying spreaders having convex upper surfaces. Means are provided for regulating the passage of hot air through the casing against the materials passing through.
F. Salt.

Production of superphosphates. B. G. Klugh, Assr. to Federal Phosphorus Co. (U.S.P. 1,583,054, 4.5.26. Appl., 7.12.20).—The process consists in passing gases containing phosphoric oxide through a mass of finely-divided materials which will react

with the phosphoric oxide and retain it, and in passing heated air through the mass to accelerate the reaction. C. T. GIMINGHAM.

Process for treating seed grain. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of A. SCHMIDT, A. STEINDORFF, A. FLUSS, and O. SCHAFFRATH (U.S.P. 1,565,237, 8.12.25. Appl., 7.4.23).—Equal parts of a 0·2% solution of the dihydrochloride of diaminodihydroxyarsenobenzene in water and of a 2% solution of copper sulphate are mixed, and the grain to be treated is immersed in this liquid for 1 hr. C. T. GIMINGHAM.

Insecticide for use on tobacco plants. H. HOWARD, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,580,200, 13.4.26. Appl., 15.5.22).—A finely divided brown-coloured material, capable of suspension in water, comprising about 2 pts. of an arsenate of manganese (containing not less than 48% of arsenic pentoxide), and about 1 pt. of burnt umber, is claimed as an insecticide. C. T. GIMINGHAM.

Procedure for destruction of the vine louse [*Phylloxera*]. J. H. HORST (G.P. 421,241, 23.12.24). —The procedure consists in a winter treatment of the vine stocks with a mixture of nitrobenzene and aniline (or benzonitrile), followed by a summer treatment with pure carbon disulphide. The pest s completely controlled and the vines are not injured. Very little carbon disulphide is required, since most of the insects are killed by the winter treatment. C. T. GIMINGHAM.

Means for eradication of animal and plant pests. E. STRICKLER (G.P. 421,833, 10.4.23).— Amyl acetate, mixed with oils, fats, waxes, resins, soaps, paraffin, vaseline, alcohol, etc., or absorbed in porous substances, such as pumice, charcoal, or infusorial earth, is claimed as a means for destroying all animal pests with which it is brought into contact. Weeds, such as plantains, are killed in 2—3 weeks by sprinkling with a dilute solution of amyl acetate. C. T. GIMINGHAM.

Procedure for combating plant diseases. A. WACKER, GES. F. ELEKTROCHEM. IND. M.B.H., Assees. of F. KAUFLER (G.P. 422,912, 5.5.22).— Cuprous halides (especially cuprous chloride) are claimed as a means for combating plant diseases. They are more easily obtainable than cupric salts, contain a higher proportion of copper and, being very slightly soluble, do not exert a corrosive action on foliage. Addition of alkalis to the spray fluid is usually unnecessary, and a higher concentration of copper can be used than with cupric salts. C. T. GIMINGHAM.

Process of making a fertiliser. E. L. PEASE (U.S.P. 1,584,130, 11.5.26. Appl., 1.4.22).—See E.P. 192,778; B., 1923, 321 A.

Producing a fertiliser of urea. J. H. LIDHOLM (U.S.P. 1,584,875, 18.5.26. Appl., 9.1.24).—See F.P. 586,025; B., 1926, 208.

Insecticide (E.P. 234,456).—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Prevention of the deterioration of raw sugars by inoculation with *Torulæ*. W. L. OWEN (Facts about Sugar; Int. Sugar J., 1926, 28, 198—203).— Deterioration of raw sugar during storage or shipment by the action of moulds is shown to be prevented by inoculation with *Torulæ*, the action of which is to restrain the attacking organism by the carbon dioxide developed within the molasses film surrounding the crystals. Sugars thus treated were found to be safer with a non-sucrose to moisture ratio of 2:1 than are inoculated sugars with a ratio of 3:1 (as prescribed by the "factor of safety"). and they are much less hygroscopic and also lighter in colour. The concentration of carbon dioxide required appears to be approximately 0·036%, which is reached generally during the first or second week after inoculation. (Cf. U.S.P., 1,579,387; following.) J. P. OGILVIE.

Use of chlorine in the refining of sugar. S. OCHI (Trans. Amer. Electrochem. Soc., 1926, 49, 147—156. Advance copy).—Mill juice at p_H 3·5—4·0 is treated with chlorine to give a chlorine concentration not exceeding 0·02*N*, the temperature not exceeding 55°, and the time being less than 30 min., after which the liquid may be filtered for the separation of the suspended matter and colloids thus coagulated, treated with a sufficient amount of activated carbon (as "Norit" or "Darco") to adsorb the excess of chlorine and the hydrochloric and hypochlorous acids, and neutralised. Raw sugar liquor (at about 40° Brix) may similarly be treated, using 0·1—0·4% of chlorine on the weight of solids in the syrup, the temperature being about 50°, but the time should not exceed 30 min., while the amount of carbon added in this case is not less than 2%. Chlorination of sugar juices and liquors is capable of removing 80—90% of the colour under the conditions specified, the bleaching effect being completed by the carbon. J. P. OGILVIE.

Precipitation of acids during defecation and carbonatation. F. PACHLOPNIK (Z. Zuckerind. Czechoslov., 1926, 50, 269—276, 281—288).— Lactic, saccharic, and hydrochloric acids were not precipitated to any extent during the defecation and carbonatation of beet juice, but phosphoric and oxalic acids were thrown down in both operations, almost quantitatively. Malic acid was eliminated in defecation only slightly, and citric acid to the extent of about 50%, though carbonatation removed the greater part of both. Hardly any sulphuric, aspartic, and glutamic acids were precipitated during defecation, but after carbonatation 74, 60, and 40% respectively passed into the scums. Salts of these acids behaved in general similarly when carbonatation was stopped at an alkalinity of 0·1%, but on continuing approximately to neutrality the quantity of acids precipitated was as a rule diminished. J. P. OGILVIE.

Decolorisation of intermediate and thick juices by "Carboraffin" and "Norit." A. LINSBAUER and J. FISER (Z. Zuckerind. Czechoslov.,

1926, 50, 225—232, 233—239, 241—247, 249—254).—Extended large-scale tests in a beet factory making white sugar showed that activated carbons applied to the decolorisation of the evaporator syrup (intermediate and thick juice) by pumping through layers of the preparation in special filters resulted in such a very small increase of purity and decrease of colour as to make the procedure uneconomical. Filtration through carbon, however, is of value in producing a bright syrup for crystallising in the vacuum pans, and for this reason the use of very cheap preparations without considering their decolorising power is advised. In the refinery carbons used as pre-filtering media may be of value in reducing the cost of operating the char station.

J. P. OGILVIE.

Rapid method for determining the loss of polarisation in beet analysis. V. STANEK and J. VONDRAK (Z. Zuckerind. Czechoslov., 1926, 50, 257—265).—75 c.c. of juice which had been clarified and "de-leaded" were heated with 5 c.c. of 20% sodium hydroxide during 15 min., cooled, neutralised with acetic acid, made up to 100 c.c., and polarised, a control observation being made with the same amount of juice in the presence of the same amount of sodium acetate. It was found in this way that the dextrorotatory substances likely thus to be destroyed may vary between 0·09 and 0·20% of the root. Their amount in beets depends upon the conditions of cultivation and growth.

J. P. OGILVIE.

Determination of water in sugar factory products by distillation with hydrocarbons. R. VYSKOCIL (Z. Zuckerind. Czechoslov., 1926, 50, 289—295).—An apparatus is described for the rapid determination of water in beet slices, carbonatation scums, syrups, massecuites, sugars, molasses, coal, bone-char, etc., by distillation with hydrocarbons. Xylene and toluene gave results with these products which uniformly were practically identical with those found by the desiccation method.

J. P. OGILVIE.

Temperature corrections to readings of Baumé hydrometers. Bureau of Standards Baumé scale for sugar solutions (standard at 20°) (U.S. Bureau of Standards, Circ. 295, 16.1.26, pp. 3).—A table is given by the use of which Baumé hydrometer readings between 0° and 40° B., made at temperatures between 0° and 80°, may be corrected to the standard temperature 20°. It is based upon Plato's values for the thermal expansion of sugar solutions (Wiss. Abh. Kaiserl. Normal Eichungs-Kommission, 1900, 2, 153). The Bureau of Standards official scale, the old or Holland, and the new or Gerlach scales are compared from 40° to 44° B. The first scale gives readings 0·1—0·2° higher than the second and 0·6° lower than the third.

G. T. PEARD.

Preparation of lævulose. R. F. JACKSON, C. G. SILSBEE, and M. J. PROFFITT (Sci. Papers U.S. Bur. Standards, 1926, 20, [519], 587—617; cf. B., 1925, 255).—Juices from the artichoke were found to contain from 8·1 to 23·8% of lævulose in the form of polysaccharides convertible by acidifying and heating to 70—80° for 30—40 min., while those from the dahlia have 9·3 to 14% of inulin, convertible to a syrup of 86—90% purity. Artichoke juices of high purity after conversion were directly crystallised, but from impure liquors the lævulose was separated as its calcium compound, which was filtered off, washed, decomposed with carbon dioxide, and the resulting solution crystallised after evaporation in a vacuum. In the process of crystallisation from aqueous solutions described, syrups with 90—92% of solids were heated to 55°, "seeded," maintained in slow movement for a sufficient time, and the mass finally centrifuged. From syrups of high purity the crystallisation proceeded so satisfactorily that the temperature could be lowered to about 25° in 24 to 36 hrs., but syrups of lower purity required 4—5 days. Crystals obtained by the authors' procedure had the form of slightly tilted cubes resembling *en masse* ordinary sugar, but adversely affected in an atmosphere of high humidity.

J. P. OGILVIE.

Oxidation products of lactose and maltose on treatment with Fehling's solution. F. HERZFELD (Z. Ver. deuts. Zucker-Ind., 1926, 177—192).—Lactose reduced Kraisy's solution (cf. B., 1921, 315 A) much less than Fehling's solution. When after separation of the cuprous oxide, the blue filtrates were acidified and boiled, and the alkalinity was restored, a greater reduction was obtained with the former than with the latter reagent. Kraisy's solution is therefore recommended in preference to the other for the examination of products containing lactose in presence of sucrose. Likewise in determining maltose the weaker copper reagent should be used, dextrin and *iso*maltose apparently being not affected by it. J. P. OGILVIE.

Analysis of lime. BAILAR.—See VII.

PATENTS.

Automatically regulating the juice level in evaporating or boiling apparatus of sugar-treating plants. NAAML. VENNOOTS. DE NEDERLANDSCH INDISCHE IND. (E.P. 235,206, 4.6.25. Conv., 7.6.24).—Juice supply or discharge is divided into a substantially constant flow for a quantity below that required for normal working, and a variable flow for a quantity determined by a controlling means, and automatically adjusted by the juice level in the apparatus. An independently arranged tank having a float is placed in communication with both the steam space and juice space of the apparatus of which the juice level is to be regulated, a by-pass connected to the juice supply or discharge conduit being regulated by a controlling device actuated by the float. The controlling device may be so constructed that between the controlling parts thereof in the closed position, there is still a small open passage. J. P. OGILVIE.

Refining of sugar juice by chlorination. S. OCHI and F. KOTERA (E.P. 249,191, 15.12.24).—

Sugar juice is chlorinated at a temperature not exceeding 55°, the excess of chlorine being removed by means of activated charcoal, and the treated juice neutralised. Chlorination may be effected by blowing chlorine into the juice, or by the electrolysis of an added chloride, (C.f. Ochi, preceding.)

J. P. OGILVIE.

Treatment of molasses and sugary juices, with a view to recovery of the sugar. C. DEGUIDE (E.P. 249,759, 15.10.25).—Monobarium silicate, or the compound, $2SiO_2,3BaO$, intermediate between this and the dibarium silicate, is added to molasses and juices for the precipitation or destruction of the impurities (in particular gums and reducing sugars), after which the sucrose is precipitated in known manner. J. P. OGILVIE.

Increasing sugar recovery [in the clarification of cane juices]. R. VACHIER (U.S.P. 1,578,220, 23.3.26. Appl., 12.5.25).—In a process of separating sugar from partially exhausted mud obtained as the residue in the clarification of cane juice, the mud is subjected to fresh water and a quantity of lime sufficient to produce coagulation and to extract the sugar content. Clear sweet-water is thus obtained, suitable as maceration water for use in the milling process. J. P. OGILVIE.

Clarifier for saccharine solutions [for use in the defecation of cane juices]. R. VACHIER (U.S.P. 1,578,221, 23.3.26. Appl., 15.10.25).—An outer shell has a central cylindrical liquid chamber with a downwardly inclined conical bottom open at its centre, a mud chamber below in the form of an inverted, hollow, truncated cone and an upper scum chamber, also in the form of a hollow truncated cone. Means are provided for supplying the liquid to be clarified to the scum chamber, and for heating it therein. A series of inverted hollow truncated baffle cones project downwards into the shell, each being provided with a cylindrical band at its lower open end, and there is a vertical adjustable overflow cone mounted in the scum chamber with means for carrying off the scum from it. Means are also provided for carrying off the clear liquid from the upper portions of the V-shaped annular spaces between the baffle cones and the outer shell. A rotary hollow perforated shaft projects up through the axis of the mud chamber, scrapers being carried by it for scraping the bottoms of the liquid and mud chambers. Water is supplied under pressure to the shaft, which is rotated, and the mud and liquid contents are removed from the bottom of the mud chamber.

J. P. OGILVIE.

Process of manufacturing beet sugar. E. E. NICHOLSON and R. B. BEAL (U.S.P. 1,578,463, 30.3.26. Appl., 6.10.24).—Raw juice is treated with an excess of calcium oxide, and then with a quantity of water-soluble aluminate to precipitate part of the excess of lime and other hydroxides present. The flocculent granular precipitate is filtered off, after which the filtrate is treated with carbon dioxide, and again filtered. J. P. OGILVIE.

Preservation of [white] sugar. W. L. OWEN; dedicated to the People of the United States (U.S.P. 1,579,387, 6.4.26. Appl., 14.9.25).—White sugar is preserved by inoculating it with *Torulæ* (cf. p. 559).

J. P. OGILVIE.

Recovering calcium hydrate [hydroxide, from calcium saccharate]. G. M. DRUMMOND, Assr. to HOLLY SUGAR CORP. (U.S.P. 1,580,024, 6.4.26. Appl., 23.9.24).—Calcium hydroxide precipitated from "cold saccharate" mixtures is recovered by introducing the mixture into positive pressure or vacuum "thickeners," from which the clear liquid flows continuously, while the precipitate is removed from the bottom, and filtered off, the filtering medium in the thickener being completely submerged throughout. J. P. OGILVIE.

Manufacture of sugar. H. SCHREIBER (U.S.P. 1,581,663, 20.4.26. Appl., 23.5.25).—Acidified beet diffusion juice is treated with pepsin, and after reaction has taken place excess of milk of lime is added and carbon dioxide is bubbled through until the alkalinity is reduced to such a point as to obtain a "break" (flocculation). The juice is then filtered and treated subsequently in the usual manner.

G. T. PEARD.

Manufacture of sugar. P. J. MCGUIRE, Assr. to DORR Co. (U.S.P. 1,583,299, 4.5.26. Appl., 25.3.20).—Sugar juice at 85—93° is limed and then, still at the same temperature, subjected to the action of carbon dioxide at a temperature between 85° and 102°. G. T. PEARD.

Decomposition of barium saccharate. W. SCHEERMESSER (G.P. 425,170, 27.1.25).—Towards the end of the process of decomposing barium saccharate by means of carbon dioxide, a direct or alternating electric current is passed through the solution, the addition of carbon dioxide, preferably under increased pressure, being continued during the passage of the current. L. A. COLES.

Manufacture of dry halogen-calcium-starch preparations. HENKEL & Co. G.M.B.H. (E.P. 244,708, 20.8.25. Conv., 16.12.24).—The addition of small quantities of borax (up to 2%) to mixtures of starch and calcium chloride (cf. G.P. 308,616; B., 1919, 56 A) confers on them the property of forming smooth pastes when mixed with water. These pastes may be used for adhesive purposes or for dressing, finishing, or stiffening fabrics. Excess of borax beyond certain limits gives undesirable properties to the mixtures. Borax in the finest possible state of division is stirred into a mixture of water, calcium chloride, and starch flour shortly before it sets, or the borax may be crushed with the solid calcium chloride and starch.

A. COULTHARD.

Manufacture of conversion products of starch. J. M. WIDMER, Assr. to PENICK & FORD, LTD. (U.S.P. 1,578,568, 30.3.26. Appl., 23.5.23).—Starch is converted into glucose in the presence of a hydrolysing acid, which is subsequently neutralised in the presence of phosphoric acid. The precipitated impurities are removed by filtration and the liquor is

treated with tannic acid, the coagulated proteins and insoluble mineral salts are filtered off, and the liquor is treated with carbon to remove excess of tannic acid and traces of colour. B. W. CLARKE.

Purification of starch. M. J. PRUCHA and J. M. WIDMER, Assrs. to PENICK & FORD, LTD. (U.S.P. 1,582,537, 27.4.26. Appl., 10.12.20).—Starch which has been separated from its vegetable source and from the bulk of the proteins, is freed from residual proteins by treating with sulphur dioxide to prevent bacterial life and then with pepsin to render the proteins soluble; the latter are then removed in solution. B. M. VENABLES.

Antifreezing solution (U.S.P. 1,581,179).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Occurrence of methyl alcohol in grapes and the products of their fermentation. J. VILAR (Anal. Asoc. Quim. Argentina, 1926, 13, 543—550).—Methyl alcohol is only formed from the skins and the solid portion of the pulp of grapes. Its production is independent of the access of air during fermentation, is favoured by an alkaline reaction, and hindered by sterilisation or an acid reaction. Methyl alcohol is formed even from skins which have been washed with water and are free from sugar. No practicable method was found for preventing the formation of methyl alcohol where fermentation takes place in the presence of skins.
G. W. ROBINSON.

Clarification of saccharine solutions [musts and wines] with mercuric salts. L. SEMICHON and FLANZY (Ann. Falsif., 1926, 19, 208—226).—Various reagents (salts of lead and mercury and animal charcoal) have been tested for their suitability for the clarification of wines and musts, preparatory to determination of sugars by reduction or rotation. Acid mercuric sulphate is the most suitable, the excess of mercury being precipitated by hydrogen sulphide. The solution is completely decolorised and remains permanently clear; owing to its acidity the reagent may cause partial hydrolysis of hydrolysable sugars such as sucrose and hence may not be used where these are present. Except in the above respect it has no effect upon the sugars present, whereas lead salts and animal charcoal may have a considerable effect. The mercury reagent removes the greater part of the optically active gums and pectins present, which the other two reagents fail to do. G. T. PEARD.

Behaviour of gelatin towards colouring matter in wines. A. KICKTON and F. MAYER (Z. Unters. Lebensm., 1926, 51, 132—137).—Gelatin-coated filter paper (cf. Van Eck, Pharm. Weekblad, 1925, 62, 365) is not a satisfactory material for detecting the presence of artificial colouring matter in wines. For this purpose the appearance of the precipitate produced by lead acetate may be of value as a preliminary test, but the most trustworthy indications are given by the wool fibre and amyl alcohol tests.
B. W. CLARKE.

Determination of aldehyde in alcoholic liquors by Schiff's reagent. K. C. BAILEY (Proc. Roy. Irish Acad., 1925, 37, B., 58—70).—The results are accurate only when the alcohol–water ratio of the sample is the same as that of the standards. The colour developed is approximately proportional to the aldehyde concentration only in liquors containing 30% of alcohol. Alcohol free from aldehyde is difficult to prepare and is unnecessary since a progressive development of colour in the test solution, upon keeping, has been noticed. Hence the final reading should be taken soon (20—25 min.) after addition of the reagent. The error due to the aldehyde in the alcohol can be calculated and allowed for. The reagent should be prepared from rosaniline base or hydrochloride and should not be yellow. A modified technique for the determination is suggested. G. T. PEARD.

Utilisation of molasses spent wash as a nitrogen-potash fertiliser. E. CERASOLI (Giorn. Chim. Ind. Appl., 1926, 8, 61—62).—In Italian molasses distilleries the spent wash is concentrated and then calcined to recover the potassium salts, which are mostly exported. In order to obviate the complete loss of the nitrogen compounds involved in this procedure, absorption of the spent wash, after concentration to 32—36° Brix (*d* 1·139—1·159) by kieselguhr or porous silica derived from the treatment of leucitic rocks is recommended. The friable, granular powder thus obtained is named kalinzote and serves as a nitrogen–potash fertiliser.
T. H. POPE.

See also A., May, 542, **Inactivation of invertase by heat** (VON EULER and JOSEPHSON); **Inhibition phenomena during enzymic hydrolysis of sucrose** (VON EULER and JOSEPHSON). 543, **Hydrogen–ion concentration and alcoholic fermentation** (HÄGGLUND, SÖDERBLOM, and TROBERG; HÄGGLUND and AUGUSTSSON); **Alcoholic fermentation by dried yeast** (ABDERHALDEN); **Formation of acetylmethylcarbinol during fermentation of sucrose by yeast** (ELION). 544, **Direct fermentation of maltose by yeast rich in maltase** (WILLSTÄTTER and BAMANN); **Synthesis and fermentation of glycogen by maltase-free yeast** (GOTTSCHALK); **Influence of previous treatment of yeast on affinity constants of invertase** (VON EULER and JOSEPHSON); **Fermentation of galactose by yeast** (VON EULER and NILSSON); **Zymase production and co-enzyme activity** (LEBEDEV); **Reductase of yeast** (VON EULER and NILSSON); **Carboligase** (BEHRENS and IVANOV). 545, **Protective action of milk yeasts on lactic acid bacteria** (SLOBODSKA-ZAYKOVSKA); **Acetone–ethyl alcohol fermentation** (BAKONYI); **Oxidation of glycerol to dihydroxyacetone by bacteria** (VIRTANEN and BÄRLAND).

PATENTS.

Recovery of ammonia from vinasses. G. P. GUIGNARD (U.S.P. 1,576,427, 9.3.26. Appl., 25.10.21).—In a process for recovering the ammonia from vinasses containing water, the product is heated under

vacuum at a temperature constantly maintained between 450° and 550°. J. P. Ogilvie.

Process of making compressed yeast. R. Kusserow (U.S.P. 1,580,500, 13.4.26. Appl., 8.5.24).—A nutrient solution for the manufacture of yeast is prepared by diluting molasses containing lactic acid bacteria with water to about *d* 1·160, and then adding at the ordinary temperature sufficient acid to acidify but not to sterilise the solution, a thiosulphate, and yeast nutrients. G. T. Peard.

Manufacturing alcohol by fermentation. H. W. Dahlberg, Assr. to Great Western Sugar Co. (U.S.P. 1,581,001, 13.4.26. Appl., 21.3.25).—A beet molasses mash, containing organic nitrogen compounds but substantially no commercially recoverable sugar, is fermented with yeast and the alcohol recovered. G. T. Peard.

Production of butyl alcohol and acetone. D. A. Legg, Assr. to Commercial Solvents Corp. (U.S.P. 1,582,408, 27.4.26. Appl., 30.3.25).—A mash containing protein-free carbohydrates and soluble nitrogenous matter derived from maize is inoculated with butyl alcohol–acetone bacteria and the products are recovered from the fermented liquor. G. T. Peard.

Liquid fuel (E.P. 249,577).—See II.

Products useful in tanning (E.P. 247,977).—See XV.

XIX.—FOODS.

Conditioning of wheat. J. S. Remington (Ind. Chem., 1926, **2**, 203—207).—Perfect conditioning of wheat, involving both physical and chemical changes, is best carried out by washing the wheat at 25—40° after centrifuging, and passing through a steam-heated jacketed worm at a temperature determined by the grade of wheat or mixture used, and subsequent mellowing for about 3 hrs. in a hot condition. After cooling and storing for a few hours the wheat is ready for milling. Unless the process is carefully controlled, the wheat is very liable to be damaged; it is important to check the moisture content at all stages of the process. Conditioning must be a slow process to develop fully the latent "strength" of the wheat and produce flour of the best quality on milling. The hydrogen-ion concentration developed in the flour during conditioning will affect the baking process. Experiments with a small-scale conditioning apparatus ("hydroliser") will indicate the optimum conditions for temperature, moisture, time, etc. for any given sample of wheat. B. W. Clarke.

Determination of the degree of milling of flour in bread. H. Kalning (Z. Unters. Lebensm., 1926, **51**, 145—147).—The ash content of bread is not a direct guide to the ash content of the flour used, which is an indication of the efficiency of milling. This is owing to the addition of salt during baking; the chlorine is mostly driven off, but the sodium is fixed in the ash as sodium carbonate, and the ash in the bread is therefore affected by the amount of salt added. A more trustworthy guide is obtained by a determination of the phosphoric acid in the ash (reckoned as P_2O_5); the ash of flour always contains 48—49% P_2O_5, and can thus be calculated from the amount and P_2O_5 content of the bread ash. This method cannot be adopted if calcium phosphate has been used as an improver during baking, as shown by a high calcium content in the ash. The ash of the bread is therefore not a very trustworthy indication of the efficiency of the milling process. B. W. Clarke.

Determination of fat in milk by the Thomé method. J. B. Platon (Z. Unters. Lebensm., 1926, **51**, 120—125).—The Thomé method (Svensk Kem. Tidsk., 1917, **29**, 148) gives slightly higher values than the Röse–Gottlieb method for the determination of fat in whole milk, skimmed milk, and buttermilk. the figure being 0·008 higher, independent of the fat content of the milk. The method differs from the Röse-Gottlieb method in that ethyl ether is used instead of light petroleum for the second extraction, and that the ether can be recovered and used again after distillation. The addition of potassium dichromate to the milk as a preservative does not affect the determination of the fat until the milk is about 3 months old. The Thomé method has the disadvantage that it is much more sensitive to the concentration of alcohol used, and is therefore more subject to error on this account. B. W. Clarke.

Determination of fat in dried milk. M. Dellepiane (Anal. Asoc. Quim. Argentina, 1925, **13**, 533—542).—The author discusses the determination of fat in dried milk by the Werner–Schmidt and Röse–Gottlieb methods. Results obtained by both methods are compared for a number of samples. Somewhat lower figures are obtained by the second method owing to slight saponification of the fat by the ammonia solution which is added at a certain stage in the procedure. G. W. Robinson.

New value in the examination of fat mixtures and butter. A. von Raalte (Chem. Weekblad, 1926, **23**, 222—224).—The "xylene-number" expresses the proportion of volatile fatty acids which remain dissolved in water when the product of steam distillation of the free fatty acids is extracted with xylene. The number is high for butter, since the lower fatty acids are little soluble in xylene, but lower for other fats, owing to the great solubility of caproic and higher acids in this medium. 5 g. of the fat are saponified, the free acids distilled in steam, 110 c.c. of distillate collected and filtered. 100 c.c. of the filtrate are shaken with 10 c.c. of water and 22 c.c. of xylene, the aqueous layer is filtered, and 100 c.c. are titrated with 0·05*N*-sodium hydroxide. The volume required in c.c.$\times$1·21$\div$2 is the xylene number. The percentage of butter in a fat is calculated from the formula (xylene number —0·63)$\div$0·23. Results of analyses of various mixtures are given. S. I. Levy.

Utilisation of chlorinated water in canning and preserving processes. H. SERGER (Z. Unters. Lebensm., 1926, **51**, 125—132).—Water purified by means of chlorine is quite suitable for use in preserving vegetables etc.; it has no effect on the flavour or appearance of preserved asparagus, carrots, or beans when used for cooking, washing, and filling purposes. No free or loosely combined chlorine can be detected in the water or the vegetables when as much as 1 g. of chlorine per cubic metre of water is used for the purification.

B. W. CLARKE.

Acids in fruits used for jam making. C. F. MUTTELET (Ann. Falsif., 1926, **19**, 235—238).—Citric and malic acids may best be determined by precipitation of their barium salts from alcoholic solution (cf. B., 1922, 726 A). The barium citrate is precipitated by the addition of ½ vol. of 95% alcohol, the malate by the addition of 2 vols. of alcohol. Malic acid is found in apples, but not in the fruits usually employed for jelly making (gooseberries, raspberries, strawberries, and currants); hence the detection of malic acid in such "pure fruit" juice denotes adulteration. In the same way adulteration of fruits containing much citric and little malic acid may be detected by a change in the ratio of the two acids. Thus the ratio is altered from 15 : 1 to 15 : 10 when cranberry juice is mixed with an equal weight of apple juice. The author recapitulates his earlier work on this subject (cf. B., 1923, 197 A).

G. T. PEARD.

Fermentation of cacao. A. W. KNAPP (J.S.C.I., 1926, **45**, 140—142 T).—Experiments on 2000 lb. of selected Trinidad cacao (mainly Forastero) show that unripe cacao does not ferment satisfactorily; it failed to reach the high temperatures normally attained, the yield was low, and the quality inferior. Over-ripe cacao ferments similarly to ripe, but the shells are fragile, and if the over-ripeness approaches germination, the flavour is spoiled. The addition of 4% of a 2½% solution of acetic acid hastened the colour changes associated with fermentation, but did not improve the final product. The addition of 0·003% of diastase (diastatic power 19·1) only slightly increased the breaking down of the pulp, and did not improve the final product. The addition of 0·3% of 3% hydrogen peroxide to the cacao did not materially increase the speed of the oxydase reaction.

Gossypol and *d*-gossypol content of some North Carolina cottonseed meals. F. W. SHERWOOD (J. Agric. Res., 1926, **32**, 793—800).—40 N. Carolina cottonseed meals contained the following amounts of gossypol and *d*-gossypol, respectively: 14 open-kettle meals, 0·021—0·150%, 0·544—0·963%; 22 continuous cooker meals, 0·007—0·228%, 0·633—1·076%; 4 "cold-press" meals, 0·02—0·102%, 0·335—0·505%. During the manufacture of the meal, 75% or more of the gossypol present in the kernels (average 0·6%) is converted into the less toxic *d*-gossypol, which unpublished data suggest is a hydrolysis product of the unstable gossypol. The toxicity of diet containing cottonseed meal is discussed. Assuming that *d*-gossypol is non-toxic, of which there is indirect evidence (cf. Schwartze and Alsberg, B., 1924, 885), only 12·5% of the above meals contained sufficient gossypol to cause injury in feeding experiments to the white rat, using a well-balanced diet containing not more than 50% of cottonseed meal.

E. H. SHARPLES.

See also A., May, 546, **Antirachitic value of irradiated cholesterol and phytosterol** (HESS, WEINSTOCK, and SHERMAN); **Diet and reproduction** (GRIJNS).

Detecting coconut oil in chocolate. RUFFY.—See XII.

PATENTS.

Insecticide or fumigant and fumigating method. R. C. ROARK (E.P. 234,456, 30.3.25. Conv., 26.5.24).—A mixture of a fatty acid ester, of boiling point not higher than 150°, with carbon tetrachloride is claimed as an insecticide or fumigant, for use especially against weevils in grain. Purified ethyl acetate is particularly suitable; a mixture of 2 pts. by volume of the acetate with 3 pts. of carbon tetrachloride is non-inflammable and inoffensive. Grain may be treated in a relatively closed space, such as a box car or bin, using about 40 lb. of the fumigant per 1000 cub. ft. of space. C. T. GIMINGHAM.

Producing a jelly base. A. GUSMER (U.S.P. 1,580,665, 13.4.26. Appl., 10.1.21).—In the preparation of a jelly base from a pectose extract obtained from vegetable pulp, a fungus possessing both diastatic and zymotic properties is added in slightly acid medium, whereby the starch and albumin constituents of the extract are modified and their precipitation is prevented. G. T. PEARD.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Preparation of lecithin from eggs. C. BARRO (Giorn. Farm. Chim., 1926, **75**, 59—61; Chem. Zentr., 1926, I., 3188).—A mixture of 180 g. of yolk of egg with washed and calcined pumice stone is dried *in vacuo* at 50°, extracted with acetone until no more colouring matter is removed, and then treated three times with about 300 c.c. of cold 95% alcohol. On evaporation of the alcohol, lecithin is obtained as a neutral, waxy solid, from which traces of fat are removed by extraction with acetone. On drying the residue *in vacuo* over sulphuric acid, 16·5 g. of lecithin are obtained; the product, however, still contains some cephalin and lutein. The factor, 11·40, corresponding to distearinlecithin, given by Moreau for calculating the weight of lecithin from its P_2O_5 content, is too high for use with egg lecithin, which consists of palmitinoleinlecithin having as factor 10·95; using this value, the product obtained as above contains 94·68% of lecithin.

L. A. COLES.

Determination of the strength of glacial acetic acid. C. O. HARVEY (Analyst, 1926, **51**, 238—239).—After determining the approximate freezing point

of the acid in a freezing mixture, a second quantity of acid is cooled about 5° below this point and supercooling allowed to take place to about 1°. The thermometer is then rapidly dipped into the first tube of frozen acid, transferred back with a few adhering crystals to the supercooled acid, and the maximum recorded temperature taken as the freezing point. For concentrations of acid between 97 and 100% by weight the relationship between concentration and freezing point is practically rectilinear and the percentage of anhydrous acetic acid may found be from the equation $x=0{\cdot}64t+89{\cdot}5$, where t is the freezing point. D. G. HEWER.

Alum as catalyst for production of ether [from alcohol]. S. K. K. JATKAR and H. E. WATSON (J.S.C.I., 1926, **45**, 168—170 T).—By means of an improved alum catalyst yields of ether about ten times as great as those previously recorded for an equal weight of catalyst have been obtained. The best operating conditions are outlined. The ether is very pure and the quantity of ethylene formed is negligible. A description is given of a unit sufficiently large to supply the requirements of a laboratory.

So-called "Western" (South Dakota) oil of American wormseed. Distillation report for 1924. Origin, nature, and physiological rôle of the essential oil. A. HOGSTAD, JUN. (Amer. J. Pharm., 1926, **98**, 188—204).—The distillation results substantiate those of the 1923 harvest (cf. B., 1925, 25), the highest yield of oil being obtained just previous to or at the time of pollination, with a gradual decrease as the plant reaches maturity. The ascaridole content of oils from the fresh plant rises, from flowering to maturity, from 2 to 62% and is increased by air-drying. Most of the oil and ascaridole is obtained in the first 5 or 10 min. of the steam distillation. Of 54 oils examined, only four satisfied the optical activity requirements of the U.S.P. and all failed to comply in relation to solubility in alcohol. A detailed microchemical study of all parts of the plant in all stages of its development is described and photomicrographs are given illustrating stages in the growth of the glandular hairs in which the oil is contained. The oil is apparently secreted by a "resinogenous" layer and serves as a protection to the plant by lowering of transpiration. The value of microchemical studies for the determination of the optimum conditions for distillation is emphasised. E. H. SHARPLES.

See also A., May, 531, **Microchemical reactions of tutocaine** (ROSENTHALER). 532, **Alkyl esters of aromatic sulphonic acids for alkylation of organic compounds** (RODIONOW). 533, **Reaction of alkaloids with furfuraldehyde-sulphuric acid** (EKKERT). 534, **Constitution of cyanomercurisalicylic acids and of hydrargyrum salicylicum** (RUPP and GERSCH). 535, **Alkalimetric determination of formaldehyde by means of sodium sulphite** (TÄUFEL and WAGNER); **Oxidimetric determination of tartaric acid in presence of nitrate** (WIKUL). 540, **Trypanocidal action of cinchona alkaloids** *in vitro* (TSAKALOTOS); **Hypnotics of barbituric acid series** (NIELSEN, HIGGINS, and SPRUTH). 541, **Pharmacology of benzyl alcohols** (CHRISTOMANOS). 547, **Ethereal oils of tobacco; Resins and aromatic substances of tobacco; Carbohydrate complex of tobacco** (SCHMUCK). 548, **Alkaloids of** ***Ceanothus americanus*** (CLARK).

PATENTS.

Manufacture of alkali salts of aromatic sulphochloramides. FARBENFABR. VORM. F. BAYER & Co. (E.P. 241,579, 17.10.25. Conv., 18.10.24).—The compounds are obtained by treating concentrated aqueous solutions of aromatic sulphonamides or their salts with bleaching powder, and simultaneously or subsequently adding an alkali salt, *e.g.*, sodium sulphate or carbonate, containing an acid radical capable of precipitating the calcium. The solution is filtered hot, *e.g.*, at 60—70°, to remove the precipitated calcium salt, and the desired sodium salt crystallises out on cooling. L. A. COLES.

Manufacture of hydrogenated cinchona alkaloids. HOWARDS & SONS, LTD., and J. W. BLAGDEN (E.P. 250,380, 24.3.25).—Cinchona alkaloids such as quinine or cinchonidine or their substitution derivatives can be hydrogenated to hexahydro-derivatives by means of hydrogen in the presence of nickel or other catalyst of this group at temperatures above 30°, and preferably at 60—80° (cf. E.P. 21,883 and 21,948 of 1914; B., 1915, 1225). The parent material may be the non-hydrogenated or the dihydro-derivative of the alkaloid in the form of base or salt and may be used dissolved in water, alcohol. or other solvent. For example, a solution of 5 pts. of quinine in 100 pts. of absolute alcohol is agitated at 20° with 3—4 pts. of a nickel catalyst in an atmosphere of hydrogen under a pressure of 20—40 lb. per sq. in. The absorption of gas ceases when the quantity of hydrogen corresponding to the production of dihydroquinine has been taken up. On raising the temperature above 40°, absorption (slower) again sets in and at about 80° double the amount of hydrogen absorbed at the first stage will have been taken up. The hexahydroquinine is isolated by evaporating the filtered solution.
A. COULTHARD.

Manufacture of methyl alcohol and formaldehyde from methane. BAKELITE GES.M.B.H., and R. HESSEN (G.P. 421,215, 9.12.22).—Methane and excess oxygen, air, or ozonised air, are heated for a short time with non-metallic inorganic porous contact material at a red heat under pressure. Immediately after the reaction, the pressure is reduced and the products (methyl alcohol and formaldehyde) are cooled and condensed by means of a stream of air or gas flowing in the opposite direction. The reaction chamber is closed with a porous or perforated plate, the openings in which are small compared with the inner diameter of the chamber. As catalysts, pumice, brick, or earthenware fragments, highly porous slag, asbestos, quartz sand, or slag wool may be used. It is advantageous to add to the

catalyst (either in layers or mixed with it) at ordinary or raised temperature, substances which take up water and give it up again at high temperatures, such as the oxides and hydroxides of the alkalis or alkaline-earths, magnesium or calcium chlorides, or anhydrous copper sulphate. A. COULTHARD.

Preparation of aminotetrazole. R. STOLLÉ and E. SCHICK (G.P. 426,343, 12.7.24).—Dicyanodiamide in aqueous solution reacts quantitatively with 2 mols. of azoimide to give 2 mols. of *C*-aminotetrazole. A. COULTHARD.

Reduction of organic compounds (G.P. 423,029).—See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Relation between time and intensity in photographic exposure. III. L. A. JONES, E. HUSE, and V. C. HALL (J. Opt. Soc. Amer., 1926, **12**, 321—348; cf. B., 1925, 972).—The failure of the reciprocity law in the case of Wratten and Wainwright panchromatic and Eastman double coated orthochromatic plates is somewhat greater than that reported previously for fast non-colour-sensitive emulsions. The panchromatic plates show a rise of 0·16 in gamma for a range of increase of intensity of one to eight million. With the orthochromatic plates there is no systematic change of gamma with intensity. Wratten slow ordinary and Eastman slow lantern plates show very great failure of the law, and also a very marked change of gamma with intensity, especially in the lower intensities. Increase of time of development of panchromatic plates results in an increase in amount of the failure, and raises the "optimal intensity." Kron's catenary formula fits the experimental results excellently for the higher speed materials and at high intensities for the materials of low speed. In general relatively fast materials, in which the average grain size is high, have a relatively low value of "optimal intensity," while slow emulsions, which have in general grains of smaller and more uniform size, have much higher "optimal intensities." Gamma is much more dependent on intensity in the case of low-speed fine-grained materials. Fast materials show only a slight variation of density for constant exposure over an enormous range of intensity, while slow emulsions give a great density variation for constant exposure. The decrease of gamma with low intensities in the case of slow emulsions is explained as due to an effective broadening of the sensitivity or size distribution, since when intensity is decreased the "optimal intensity" of the bigger, more sensitive grains is approached, while that of the less sensitive, smaller classes is departed from; the more sensitive grains thus become relatively more effective, and the less sensitive grains relatively less effective in contributing to density. W. CLARK.

Neocyanine: a new sensitiser for the infra-red. M. L. DUNDON, A. L. SCHOEN, and R. M. BRIGGS (J. Opt. Soc. Amer., 1926, **12**, 397—400).—Neocyanine, a new sensitiser obtained first during the manufacture of Kryptocyanine, and less soluble than the latter, sensitises more powerfully than any other known dye at wave-lengths greater than 800$\mu\mu$. The sensitised band stretches from 700$\mu\mu$ to 900$\mu\mu$ with a maximum at about 830$\mu\mu$. For the mercury line at 1014$\mu\mu$, Neocyanine-sensitised plates require one-fifth the exposure of plates sensitised with Dicyanine, and the line at 1128·8$\mu\mu$ is easily photographed with long exposures. The dye gives excellent results by bathing or in the emulsion, and plates can be hypersensitised with ammonia. W. CLARK.

Colour sensitivity of photographic materials. C. E. K. MEES (J. Franklin Inst., 1926, **201**, 525—551).—An account is given of work on the normal colour sensitivity of emulsions, and the variation of gamma with wave-length; sensitising by dyes and other means is dealt with somewhat fully, and certain practical applications of sensitised materials are discussed. W. CLARK.

PATENTS.

Photographic developer. P. SCHESTAKOFF and B. MEREJKOVSKY (F.P. 600,532, 21.4.25).—Salts of acids containing nitrogen, hydroxylamine, salts of sulphonic acids of hydroxylamine, and similar compounds are added to the developer to act against the effects of under- and over-exposure, fog, and similar faults. W. CLARK.

Development in bright illumination and daylight. K. BINDER (F.P. 600,801, 15.7.25).—Complex iron phenolic compounds, especially alkali salts of the tripyrocatechol–ferric complex, are added to the developer. The plate is immersed in the developer in red light, after which any convenient illumination can be used. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Preventing or quenching explosions or fires in mines and other places. WASHINGTON CHEMICAL CO., LTD., and G. S. NEWALL (E.P. 250,739, 26.3.25).—The explosion of a blasting charge causes the dispersion of dry pulverulent basic magnesium carbonate enclosed in containers or cartridges inserted in the shot-hole in close association with the blasting charge, producing a zone of carbon dioxide, water vapour, and non-combustible powder sufficiently large to prevent or quench explosion or fire due to ignition of coal dust. J. S. G. THOMAS.

XXIII.—SANITATION; WATER PURIFICATION.

See A., May, 515, **Preparation of 2:4-dihydroxydiphenylmethane and of 2:4-dihydroxydiphenylethane** (KLARMANN). 516, **Alkyl and aryl derivatives of phloroglucinol** (KLARMANN and FIGDOR). 541, **Effect of hydrogen-ion concentration on antiseptic action of certain phenols and aromatic acids** (KURODA).

PATENTS

Treating waste liquors containing cyanogen compounds (E.P. 250,824).—See VII.

Fumigant (E.P. 234,456).—See XIX.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 23, 1926.

I.—GENERAL; PLANT; MACHINERY.

Rôle of graphite in lubrication. F. L. Koethen (Ind. Eng. Chem., 1926, 18, 497—499).—In experiments on a Riehle friction-testing machine, the pressure at which film lubrication broke down was 80% higher when using graphite mixed with oil than with oil alone. A substantial reduction in friction as indicated by the sliding weight method was also shown with graphite. The graphite film fills in the inequalities of the surface, but it is doubtful if any graphite is adsorbed in the metal. Preferential adsorption of graphite by oil, rather than by water, makes a graphited surface desirable in steel rolling-mill necks, which are subjected to the action of water. W. N. Hoyte.

Device for circulating fluids under high pressure. I. R. McHaffie (Trans. Faraday Soc., 1926, 21, 147—149).—See B., 1926, 255.

See also A., June, 569, **Relations between temperatures, pressures, and densities of gases** (Pickering). 593, **Extraction apparatus** (Brüggmann).

Determination of hygroscopicity. Gericke.—See XVI.

Patents.

Separation of the constituents of gaseous mixtures. C. C. Van Nuys and W. Wilkinson (U.S.P. 1,571,461, 2.2.26. Appl., 24.6.24).—The cold outgoing gases are caused to exchange heat with the compressed incoming gases. A portion is liquefied and used to cool the main body of gas, the temperature of which has been lowered by compression and partial expansion, by mixing and rectifying, the liquefied gases serving not only as make-up liquid but also as reflux liquid. E. S. Kreis.

Dephlegmator. G. Egloff and H. P. Benner, Assrs. to Universal Oil Products Co. (U.S.P. 1,582,588, 27.4.26. Appl., 2.5.21).—The column contains a number of vertically spaced perforated plates, the distance between which is adjustable. S. Bowman.

Still. J. Young (U.S.P. 1,584,267, 11.5.26. Appl., 17.1.22).—The vapour outlet of a still slopes upwards and is provided with a number of plates depending from the upper wall and extending part of the way across the conduit, and a number of perforated plates upstanding from the bottom, alternating with the other plates. B. M. Venables.

Apparatus for treating liquid with gas. W. E. Greenawalt (U.S.P. 1,583,141 and 1,583,591, 4.5.26. Appl., [a] 21.3.24, [b] 29.6.22).—(a) Instead of the gas being blown up from the bottom of the chamber through perforations in the supply pipes or through porous bodies placed at the bottom of the tank, as is usually the practice in the flotation of ores, the gas passes into a pipe, open at the bottom for the intake of liquid, which has at its head a mixing chamber, also beneath the surface of the liquid, where the gas and liquid are thoroughly mixed, and from which they pass out through curved radial discharge passages into the body of the liquid (cf. U.S.P. 1,374,500; B., 1921, 395 a). (b) The liquid is treated in a tank of suitable depth, the gas being blown up into a pipe, open to the liquid at its lower end, and having at its upper end, submerged beneath the surface of the liquid, a hollow rotary member with inclined discharge passages, the peripheral wall of the rotary member being continuous and cylindrical. The gas and the liquid pass up into the interior of the rotary member and are there mixed, then passing tangentially into the surrounding liquid. E. S. Kreis.

Apparatus for drying and the like. J. B. Welch (U.S.P. 1,583,376, 4.5.26. Appl., 5.12.24).—In drying operations where the temperature and humidity are varied as the condition of the material being dried alters, the conditions are altered automatically by means of electromagnetic controls operated through a series of contacts over which an arm moves, its movement being controlled by the changes taking place in a sample specimen of the material being dried, which is situated in the actual drying chamber under the same conditions as the main mass of material. E. S. Kreis.

Refrigeration. C. L. A. M. Leblanc, Assr. to Soc. Anon. pour l'Exploit. des Proc. Maurice Leblanc-Vickers (U.S.P. 1,584,382, 11.5.26. Appl., 22.9.21).—A body of gas coming from a refrigerator at a comparatively high temperature is compressed and further heated, and is then displaced to a cooling device by a second body of gas from the same cooling device at the same pressure, but at a lower temperature. The second body of gas is expanded to cool it and displaced to the refrigerator by a third body of gas coming from the refrigerator. The third body of gas is compressed and displaced to the cooling device by means of the first body of gas. The first body of gas is then expanded and displaced to the refrigerator by the second body of gas. B. M. Venables.

Drying mechanism. S. GERTZ, Assr. to THE C. O. BARTLETT & SNOW CO. (U.S.P. 1,584,764, 18.5.26. Appl., 26.10.22).—The material is treated in a rotary cylinder mounted within a heating chamber, and the vapours or gases evolved are withdrawn through a stationary longitudinal duct mounted within the cylinder near its upper wall and provided with shielded spaced openings.
H. HOLMES.

Adsorbent material. A. S. BEHRMAN, Assr. to INTERNATIONAL FILTER CO. (U.S.P. 1,584,716, 18.5.26. Appl., 1.10.24).—A solution of a salt of an amphoteric metal is mixed with a solution of an alkali silicate in proportions and concentrations to produce a gel which is acid to methyl orange and includes all the contents of the solutions. This gel is dried and leached to remove the soluble constituents. H. HOLMES.

Crusher. A. H. STEBBINS (U.S.P. 1,585,052, 18.5.26. Appl., 15.6.22).—A hollow upright shaft is suspended from an upper bearing for gyratory rotation within a casing and carries a crushing surface co-operating with the inner wall of the casing. The gyratory movement is obtained by the rotation of a second shaft mounted within the hollow shaft and carrying unbalanced weights.
H. HOLMES.

Filter. O. HOY (U.S.P. 1,585,246, 18.5.26. Appl., 29.1.23).—An inverted filter bag is mounted within a casing with its edges seating around an inlet in the bottom of the casing, and held in position by an openwork frame surrounding the bag. The bag is larger than the frame and protrudes through the openings therein. A discharge outlet is provided for the casing. H. HOLMES.

Machine for continuous separation of solids from liquids or liquids from solids. W. C. LAUGHLIN, Assr. to LAUGHLIN FILTER CORP. (U.S.P. 1,585,393, 18.5.26. Appl., 21.6.23).—Two smooth-surfaced rotary dished plates are mounted with their outer edges held in smooth contact by the pressure of a spring bearing centrally on one plate, and a baffle is mounted in the space between them. Means are provided for continuously delivering the mixture axially into this space and for discharging a portion thereof at or near the axis, while the separated heavier constituent separates the edges of the plates against the spring pressure. H. HOLMES.

Tunnel kiln (U.S.P. 1,584,883 and 1,585,013).—See VIII.

Heating liquids (E.P. 242,253).—See XI.

Calcining lithopone etc. (U.S.P. 1,584,381).—See XIII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

The Lancashire coalfield. The King Seam (Physical and Chemical Survey of the National Coal Resources. No. 6, pp. 33).—The King seam is traceable throughout most of the Lancashire coalfield, but its quality and thickness vary, and it may split into two or more seams. At its best, it is usually divided by a shale or dirt band of varying thickness. It is composed of bituminous and moderately caking coal, used mostly for manufacturing, and to a lesser extent for industrial and gasworks purposes, and as a bunker fuel. The volatile matter is rather high, and generally higher near the top of the seam. The ash is often very high, due to dirt bands and high inherent ash. The nitrogen is rather high, and the sulphur variable but rather high. Cannel sometimes occurs at the top of the seam. R. A. A. TAYLOR.

Some analyses of peat in the light of recent theories of coal formation. S. ODÉN and S. LINDBERG (Brennstoff-Chem., 1926, 7, 165—170).—The lignin, cellulose, humic acids, and waxes and resins in samples of peat of different ages have been determined (cf. B., 1926, 82). With increasing age there is a decrease in the lignin content of the peat, but an approximately proportional increase in the humic acids. Both cellulose and waxes and resins decrease in amount with the age of the peat. The results are in accord with the "lignin theory" of coal formation (cf. Fischer, B., 1924, 584). Similar changes occur when peat is subjected to bacterial action. An attempt is made to illustrate the transformation from lignin to humic acid, based on Klason's formula for lignin and Eller's work on the humic acids. The constitution deduced for humic acid gives an equivalent weight of 352, in good agreement with the values found for the natural product.
A. B. MANNING.

Base exchange and its bearing on the origin of coal. E. M. TAYLOR (Fuel, 1926, 5, 195—202).—When soil is exposed to sodium chloride solutions, base exchange takes place with the formation of a sodium absorption complex which on the removal of the neutral sodium salt becomes impermeable to water and gases and is capable of hydrolysis yielding an alkaline solution. Vegetable material below such a medium would undergo anaerobic decomposition in an alkaline medium. Investigation of decomposing vegetable material in the Nile Delta has shown the existence of such conditions, and an examination of the material revealed the presence of a substance closely resembling the fusain of coal. The majority of roofs of coal seams from typical British coalfields are alkaline and contain a large quantity of replaceable sodium, showing that base exchange has occurred either during or after deposition. It is suggested that coal has been produced by two types of bacterial decomposition. The first type, which was not truly anaerobic, resulted in the production of peat, the acidic products accumulated, and bacterial decomposition was inhibited. The second type proceeded under alkaline anaerobic conditions beneath an alkaline layer of clay produced as the result of base exchange between clay and solutions of sodium chloride. These conditions would account for the elimination of oxygen as the vegetable material decomposed, and the product of the decomposition

of peat under these conditions would be coal substance. A. C. Monkhouse.

Composition of durain. N. Simpkin (J.S.C.I., 1926, 76—78 t).—Durain, from a variety of Lancashire seams, contains 1·16% to 28% of ash, which is generally white or very pale brown and melts above 1400°, even though the coal from which it is obtained gives a readily fusible ash. In some cases the ash content is less than that of the remainder of the seam. The content of volatile matter varies considerably, as does the nature of the coke produced at 900°, but the agglutinating value is lower than that of the remainder of the coal. The cal. value compares favourably with that of the whole seam sample, and the sulphur content (generally 0·5—0·7%, apparently organic) is much less than that of the rest of the coal. When carbonised at low temperatures durain gives varying grades of cokes, some non-coherent, others hard and strong; the yield of liquor is often high, but the gas yield is usually lower than from the remainder of the seam. It is almost impossible to obtain a good separation of mineral matter from durain, as it is in a fine state of division and almost homogeneously distributed. W. T. K. Braunholtz.

Examination of clarain or anthraxylon in coal. E. Stach (Glückauf, 1925, 61, 1398—1402; Chem. Zentr., 1926, I., 2759).—A microscopical examination of sections affords no information as to the parent substance of clarain, and the methods of Gümbel (incineration) and of Turner and Randall (surface incineration of a section of coal and microscopical examination by reflected light) are more suitable. These latter methods indicate that the pitch-coal ("Pechkohle") of Upper Bavaria is a humus coal and that the woody material plays a leading part in the formation of clarain. W. T. K. Braunholtz.

Cleaning of coal. II., III. W. R. Chapman and R. A. Mott (Fuel, 1926, 5, 184—194, 245—262; cf. B., 1926, 473).—Various washing tests to aid the choice and control of coal washing plant are reviewed. The "float and sink" method using graded sizes of coal and the Henry tube method are both commended, the latter being more suitable for the construction of curves. The *X*-ray method (cf. B., 1925, 656) is criticised owing to its complexity of operation and its uncertainty with regard to durain bands in the coal. A mathematical analysis is made of the factors involved in the wet separation by density of coal from dirt and its application to practice. A. C. Monkhouse.

Primary decomposition of coal. S. Uchida (Fuel, 1926, 5, 221—224).—Samples of vitrain and durain from the Parkgate (Yorkshire) seam were distilled in an inverted vertical retort fitted with a condensing vessel for tar and connected through a glass-wool scrubber to a Sprengel mercury pump. Curves showing the gases evolved from each coal are given for every 50° from 100° to 600°. Carbon dioxide, carbon monoxide, and water vapour were evolved at a lower temperature from durain than from vitrain. The main decomposition occurring above 350° followed a similar course for both vitrain and durain, the production from durain being less than from vitrain (cf. B., 1919, 614 a). The tar yields were as follows: vitrain 10·4%, containing neutral oils 55·8%, alkali-soluble 35·6%, acid-soluble 1·2%; durain 9·7%, containing neutral oils 83·8%, alkali-soluble 3·4%, acid-soluble 9·3%. The low content of phenols in durain tar is in accord with the work of Pictet (B., 1913, 1098). The bromine absorption value of the two coals obtained by treating the sample for 24 hrs. with a solution of bromine in carbon tetrachloride was 10% for vitrain and 10·8% for durain. A. C. Monkhouse.

Transient fusion of coal. E. Audibert (Fuel, 1926, 5, 229—244; cf. B., 1926, 225).—The temperatures of decomposition, as indicated by a loss in weight, of a number of French coals, were determined and a comparison was made with the softening points using the method of Charpy and Durand (B., 1921, 72 a). Coals having a temperature of decomposition below the temperature of fusion lose their property of fusibility if heated for a sufficient length of time below their softening point; this was confirmed by microscopical examination of the coke. Comparisons are made with the fusion of potassium chlorate and rhombic sulphur. Coals which melt completely when heated at a rate not greater than half a degree per minute yield a satisfactory metallurgical coke when carbonised in coke-ovens, but coals which do not coke under these conditions will yield satisfactory cokes when either rapidly heated to a temperature slightly above their softening point and then further heated at the normal rate or by admixture with coking coals prior to carbonisation. A. C. Monkhouse.

Combustion of volatile matter in [boiler] furnaces with mechanical grates. V. Kammerer (Bull. Soc. Ind. Mulhouse, 1926, 92, 111—133).—The heat liberated by the combustion of the fixed carbon in coal is directly proportional to the content of this constituent, whereas that liberated by the combustion of the volatile matter is not proportional to the content of the latter. Coals with a low content of volatile matter yield gas which is rich in hydrogen and poor in oxygen and with a high cal. value, whilst coals with a high content of volatile matter yield gas rich in oxygen and of lower cal. value. The combustion space between grate and water-tubes must be large enough to permit of complete combustion of the volatile matter, failing which secondary air should be introduced near the top of the combustion chamber; and in practice the nature of the coals used and the rate of firing must be considered. W. T. K. Braunholtz.

Thermal analysis of carbonisation. H. Winter (Brennstoff-Chem., 1926, 7, 117—123).—Two similar plates of the substance to be carbonised (compressed, if necessary, as in the case of coal) are laid alongside but insulated from two similar plates of porcelain

in an atmosphere of nitrogen in an electric furnace. Thermocouples are introduced so as to enable the temperature of the substance and any temperature differences between the substance and the porcelain, due to endothermic or exothermic reactions, to be measured, so that a series of observations can be made of the temperature effects every 10° and the time taken for the temperature to rise 10°. If the results are plotted, changes in the temperature effects are represented by changes in direction of the curves, and the various stages in carbonisation (*e.g.*, liberation of superficial moisture, reaction water, gases, etc.) can be followed. The carbonisation of beech wood is endothermic up to 180°, at which temperature reaction water is liberated and exothermic effects predominate. A lignite (" Gagat ") with 61·4% of volatile matter, began to evolve reaction water and gases at 220° and exothermic reactions became predominant above 280°. Cannel and coking coals, with 30% and 21% of volatile matter respectively, began to evolve water at 180—200° and endothermic reactions predominated up to 250°. Between 250° and 300° thermal equilibrium existed. Exothermic effects then preponderated up to 450°, when there was a further absorption of heat, presumably required for the decomposition or distillation of the bituminous constituents of the coal. Above 470°, however, the reactions were exothermic. Coke itself gives only slight temperature effects, these being mainly due to a small amount of volatile matter present in it. W. T. K. BRAUNHOLTZ.

Thickening of benzol wash-oil. III. Valuation of benzol wash-oil in coking practice. R. KATTWINKEL (Brennstoff-Chem., 1926, 7, 123—124).—If the oil, after dehydration with calcium chloride, is fractionated to give two fractions of b.p. 200—250° and 250—300° respectively, the ratio % 250—300° : % 200—250° (termed the " thickening quotient ") bears a linear relation to the Engler viscosity of the oil, though the slope of the straight line connecting them will vary from plant to plant. Determinations of the viscosity of the oil, by means of the " thickening quotient," give a more trustworthy valuation of the wash-oil than determinations of the total distillate between 200° and 300°, and it may be reckoned that the oil should be changed when its viscosity reaches about 15° Engler at 20°. W. T. K. BRAUNHOLTZ.

Synthetic fuel from carbon monoxide and hydrogen. O. C. ELVINS and A. W. NASH (Fuel, 1926, 5, 263—265).—The catalyst used is prepared from mixtures of hydrated oxides of copper, cobalt, and manganese. These are dried at 150° and then reduced in a current of pure hydrogen below 400°; the copper oxide lowers the temperature of reduction. A mixture of equal volumes of carbon monoxide and hydrogen is passed over the catalyst at a rate of 6 litres per hr. with a catalyst volume of 140 c.c. The carbon dioxide in the escaping gases rose from 4·3% at 245° to 10·7% at 284°. The gases after cooling pass through active charcoal which by subsequent treatment and distillation yields an oil stable to light, of composition C 84·1%, H 14·1%, and boiling range 45—105°. A. C. MONKHOUSE.

Suitability of oils of high phenol content, especially low-temperature tar oils, for the operation of Diesel engines. A. SPILKER (Brennstoff-Chem., 1926, 7, 170—173).—The presence of a high percentage of phenols in a fuel oil lowers the cal. value slightly, but the prejudice against the use of such oils in Diesel engines on account of their possible corrosive action is unfounded. This has been proved by a three-months test on a four-cylinder, 500 h.p. Diesel engine, using an oil derived from a low-temperature tar, and containing 40% of phenols. No trace of corrosion could be detected, nor was there any sign of the formation of resinous polymerisation products or of carbon deposition. Corrosion, when it occurs, is due to the presence of sulphur compounds in the oil, not to the phenols. Removal of the unsaturated hydrocarbons from the light oil of low-temperature tar by sulphuric acid washing is not an economic process; the more rational method of converting this oil into a suitable motor spirit is by hydrogenation. A. B. MANNING.

Determination of unsaturated, aromatic, naphthene, and paraffin hydrocarbons in motor fuels and their automotive equivalents. G. EGLOFF and J. C. MORRELL (Ind. Eng. Chem., 1926, 18, 354—356).—The fraction to 210° from the distillation of motor fuel in a Hempel column was selected for analysis. Unsaturated hydrocarbons are determined by agitating the oil (500 c.c.) with two volumes of 80% sulphuric acid, and noting the decrease in volume. After neutralising and washing with water, the acid-treated oil is refractionated up to 210°, the residue representing unsaturated hydrocarbons that have been polymerised by the acid treatment. To determine the aromatic hydrocarbons, 20 c.c. of the fraction are nitrated with 50 c.c. of a mixture of 25% of nitric acid, 58% of sulphuric acid, and 17% of water, with which mixture the nitration products separate as a distinct layer; the content of aromatic hydrocarbons (in the second fraction to 210°) is found by multiplying the number of c.c. of nitration product by 4·3. Naphthene hydrocarbons are determined by the aniline cloud test of Tizard and Marshall (J.S.C.I., 1921, 20 T), and the content of paraffin hydrocarbons is obtained by difference. The method of analysis tested on synthetic mixtures gave fairly concordant results. The content of aromatic hydrocarbons in cracked gasolines was calculated by the aid of Ricardo's data for toluene. As an antidetonating agent 5% of unsaturated hydrocarbons or 4% of naphthenes is equivalent to 1% of toluene. Tables are given showing the highest useful compression ratios of petrol motors and the relation between b.p. and sp. gr. The results of tests on motors gave results confirmatory of the analytical data. H. MOORE.

Antiknock materials. W. H. CHARCH, E. MACK, JUN., and C. E. BOORD (Ind. Eng. Chem., 1926, 18, 334—340).—The effectiveness of antiknock compounds was studied on a $\frac{3}{4}$ h.p. Delco motor

with bouncing pin appliance, that of lead tetraethyl being taken at 100, and that of other compounds as inversely proportional to the number of molecules giving the same effect as 1 mol. of lead tetraethyl in a given volume of the same fuel mixture. Nickel carbonyl has a coefficient of 30, bismuth triaryl and trialkyl compounds 18·2 to 20·2. The valency of an element is not determinative of the effectiveness of its compounds. Compounds which suppressed the detonation of acetylene mixtures in an open tube often had no effect in a motor using kerosene as fuel, and no compound tested affected the detonation of hydrogen–air mixtures in an open tube. Detonation appears to be a sudden disruption of the molecule, and depends on its stability towards heat. Straight-chain compounds detonate more readily than ring compounds, and long chains more readily than the lower members of the same series. The detonation tube is not a satisfactory means of studying the behaviour of antidetonators in the engine. Intensity of detonation is proportional to the electric conductivity or ionisation of the cylinder gases during combustion. Organo-metallic antiknock compounds on heating deposit the finely divided metal more rapidly than they undergo oxidation. The metal undergoes oxidation with rise in temperature, and acts as an auxiliary source of ignition. Compounds with small or negative heats of oxidation have no antiknock effect. An effective antiknock compound is decomposed simultaneously with the firing of the fuel, and initiates an auxiliary area of combustion in advance of the flame front. Compounds in which lead is bound to oxygen have no antidetonating properties. A high heat of combustion accompanies antiknock action. Bismuth alkyls are decomposed at temperatures little over 100° and produce high cylinder pressures before firing, while acting as antidetonators. The antiknock effect of iodine is explained by its action as an oxidation catalyst. H. Moore.

"Motyl" and "motalin." [Iron carbonyl as antiknocking agent.] W. Ostwald (Petroleum, 1926, **22**, 525—530).—"Motalin," recently introduced as an anti-knock fuel, is a motor fuel containing 0·4% of "motyl," which is a mixture of equal volumes of iron carbonyl and benzine. On exposure to light "motalin" deposits a fine brown precipitate, the separation of which diminishes the anti-knock properties of the fuel. The iron oxide produced by the combustion of "motalin" (1·14 g. per litre) is mainly blown out through the exhaust pipe and may lead to blockages in the silencer. In a test run little oxide was deposited on the cylinder head, but sparking-plug points were affected and the exhaust-valve seatings became covered with a ferruginous deposit. The lubricating oil was found to be impregnated with finely divided iron oxide. A cumulative mud deposit in the oil appears to be inevitable. W. N. Hoyte.

Motor carbon deposits formed under controlled conditions from automobile oils. C. J. Livingstone, S. P. Marley, and W. A. Gruse (Ind. Eng. Chem., 1926, **18**, 502—504).—Tests on a small single-cylinder motor showed that Coastal oils gave smaller carbon values (grams of carbon deposited per litre of oil fed) than Mid-continent oils, and these in turn on the average gave smaller values than Pennsylvanian oils; average values were 3·8, 5·4, and 7·5 respectively for 15-hour runs. The carbon deposited from Coastal oil contained less oil than that from Pennsylvania and Mid-continent oils. The carbon values obtained are closely related to the Conradson carbon residues of the oils. In general for oils of the same viscosity that possessing the lowest boiling range gives the smallest carbon value. W. N. Hoyte.

Use of equilibrium oils for automotive engines. R. E. Wilson and R. E. Wilkin (Ind. Eng. Chem., 1926, **18**, 486—490).—The bad effects of crank-case dilution in internal-combustion engines can be most easily overcome by introducing into the lubricating oil, before charging it to the engine, an artificial diluent corresponding in amount and composition with the diluent which would eventually be present in the oil when a state of equilibrium is reached. The average diluent found in crank-case oils boiled between 135° and 230°, with 50% volatile at 175°. The satisfactory equilibrium mixture was taken as having a viscosity of 180 to 200 sec. at 38°, and was produced from oil having a viscosity of 480 sec. at 38° with 10·5% of diluent added. This equilibrium oil is found to have an abnormally low temperature coefficient of viscosity compared with straight lubricating oils. Service tests carried out with this oil indicate its superiority to oils in present use. The fundamental laws governing dilution are considered mathematically. W. N. Hoyte.

Comparison of lubricating efficiencies of oils and some of their physical and chemical properties. M. V. Dover (Ind. Eng. Chem., 1926, **18**, 499—501).—Three mineral oils (fairly light lubricants) and one vegetable oil (olive oil) were examined. For the mineral oils the connexion between low inter-facial tension (oil and water) and low static coefficients of friction did not hold; also a high iodine value for the mineral oil did not correspond with a low interfacial tension. Experiments on the flow of a turbine oil through metallic capillaries, whereby it was hoped that some light might be thrown on oiliness or the tendency to form films, gave very erratic results. Data are given for the static coefficient of friction, interfacial tension, surface tension, flash point, and acid and iodine values of the oils. W. N. Hoyte.

Oxidation of petroleum oils. G. S. Petrov and A. I. Danilovich (Trans. Karpov Inst. Chem. [Russia], 1925, [4], 143—156; Chem. Abstr., 1926, **20**, 1512).—Distilled Caucasian oil, mixed with fuming sulphuric acid, when submitted to the action of a current of air at 140° for 40 hrs., gave a reddish-yellow oil having acid value 22·14, saponification value 61·58, bromine value 8·59, iodine value 11·09. The yield of acids was 5·20% of the original oil, The purified acids had saponification value 219·0. acid value 118·0, iodine value 14·4, acids soluble in

benzene 60·1%, acids and resins insoluble in benzene 39·1%. Hydrocarbon oils when heated in a current of air in the presence of cobalt naphthenate gave a maximum yield of 8·7% of acids. The presence of hydroxy-acids insoluble in benzene is detrimental to the quality of the soap afforded. A. A. Eldridge.

See also A., June, 572, **Adsorption of carbon dioxide by activated coconut charcoal** (Rowe); **Physical properties of active charcoals** (Herbst). 595, **Oxidation of organic substances by copper oxide** (Steopoe).

Eliminating sulphur impurities from petroleum. Stadnikov and others.—See III.

Ammonia from coke-oven gas. Fokin.—See VII.

Preparation of hydrogen from water-gas. Cicali.—See VII.

Patents.

Transformation of crude peat into a coal-like substance. O. Linker (U.S.P. 1,580,058, 13.4.26. Appl., 23.7.25).—The peat mass is finely disintegrated in the presence of water containing a salt (*e.g.*, ferrous sulphate) to assist filtration, and the mixture conveyed through series of filtering cells, in the last series of which it remains until hard enough to be handled. It is then removed and submitted to the action of steam in a chamber substantially free from uncombined oxygen. W. T. K. Braunholtz.

Production of fuel mixtures. T. H. Butler, H. W. Robinson, and D. W. Parkes (U.S.P. 1,583,573, 4.5.26. Appl., 21.12.25).—A mixture of pitch and fuel oil is obtained as the residue from the distillation of the fuel oil with coal tar. W. T. K. Braunholtz.

Preparation of coal dust for the production of a coal of low ash in granular form. Soc. de Recherches et de Perfectionnements Ind. (G.P. 425,701, 1.4.23).—In the preparation of a coal conglomerate by the Trent process (cf. Perrott and Kinney, B., 1921, 615 a), an oil is used which is comparatively fluid during the stirring process but later becomes very viscid. It is added in the proportion of 1 pt. of oil to 4 pts. of coal. The conglomerate produced is in definitely separated grains, of about the size and hardness of grains of corn, which give very little dust on rubbing together. A. Coulthard.

Agglomerating coal. F. Seidenschnur and H. Pape (G.P. 427,271, 5.7.24).—Coal is agglomerated into lumps by passing through it for a short time hot inert gases in such quantity that volatile constituents of the coal, especially those capable of forming tar, are rapidly decomposed, forming a deposit of carbon, which binds the particles together. L. A. Coles.

Combustion, gasification or degasification of pulverulent solid or atomised liquid fuels. G. Szikla (E.P. 228,906, 4.2.25. Conv., 4.2.24).—The fuel is held in suspension in an ascending gas current in a downwardly tapering shaft, into which it is introduced in the vicinity of the narrowest cross-section and above the point of introduction of the gas (*e.g.*, air, steam). An enlarged chamber, arranged beneath the lower end of the shaft, is provided with draught inlets, a grate for collecting fuel particles descending through the shaft, and a water seal from which the ash is mechanically removed. The gas supply is periodically partly or entirely cut off to enable ash and unconsumed particles of fuel to fall out of the reaction zone, the supply of fuel being simultaneously reduced or cut off. W. T. K. Braunholtz.

Gas manufacture. G. Helps (E.P. 250,626, 17.11.24).—The plant consists of a vertical retort superimposed on a generator. A portion of the hot producer-gas, or "blow" gas if water-gas be made, from the generator passes through nostrils and around the retort either to the stack or to a waste-heat boiler. The calorific value of the gas made can be varied by allowing a portion of the producer gas to pass through instead of around the retort to mix with the distillation gases. (Reference is directed, in pursuance of Sect. 7 (4) of the Patents and Designs Acts, 1907 and 1919, to E.P. 207,851, 165,456, 164,935, 151,445, and 4572 of 1913.) A. C. Monkhouse.

Fuel distilling apparatus. Distillation of solid fuels and other substances. Merz and McLellan, and E. G. Weeks (E.P. 251,051, 29.4.26. Appl., [a] 25.2.25, [b] 13.11.25.—(a) An extracting device for removing coke from a vertical retort in such a way that the fuel moves uniformly through the retort is described. It consists of a number of rollers built up of separate cog wheels, each roller revolving in a separate compartment but all discharging material into one common coke chamber. (b) The coke extractor described in (a) is so modified that the direction of rotation of any roller can be periodically reversed. In this way parts of the fuel column undergoing carbonisation are quiescent whilst those parts sandwiching it are in motion. When reversal of the extractor rollers takes place the zones previously quiescent come into motion and *vice versa*. S. Pexton.

Distilling carbonaceous materials. M. J. Trumble (E.P. 251,142, 29.4.26. Appl., 29.2.25).—Superheated steam is partly decomposed into hydrogen by passage over some readily oxidisable material heated in a furnace. The excess steam together with the hydrogen produced is introduced into a cylindrical metal retort, externally heated, containing the oil shale or similar material to be distilled. The distillation products pass through a baffle within the retort to remove solid particles and the oil is then separated from the gas by condensation. S. Pexton.

Distillation or coking of fuels. J. Plassmann (E.P. 251,508, 11.9.25).—The fuel is heated in narrow vertical chambers with movable side walls and bottom; the gas is withdrawn through the

side wall into a chamber at the side of the retort beds. The coke is discharged into a bunker filled with inert gas which during carbonisation is at the same as, or a higher pressure than, the carbonisation gases. The coke is cooled by the circulation of the gases, saturated with water, in the bunker.

A. C. MONKHOUSE.

Dry cooling of coke. R. WUSSOW (E.P. 251,530, 22.10.25).—The cooling chamber is connected at its lower half with a small chamber which acts as a steam saturator. By means of a fan, gas saturated with steam is withdrawn and passes up through the hot coke; the heated gas returns to the top of the saturator where it meets a spray of water. The excess steam passes through the upper portion of the coke in the cooler and is converted into water-gas. The process may be used in conjunction with a vertical retort in which case air or oxygen is admitted to burn the water-gas to give sufficient heat for the carbonisation of the coal.

A. C. MONKHOUSE.

Coke from mixtures of solid and less volatile liquid fuels. G. BAUME (F.P. 596,191, 11.7.24).—The mixture, together with catalysts, is distilled in coke ovens.

A. COULTHARD.

Improvement of coke. L'AIR LIQUIDE (SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE) (F.P. 597,150, 6.8.24).—After coking has taken place, a stream of gas rich in methane or other hydrocarbons which easily dissociate, is led over the still glowing coke.

A. COULTHARD.

Drying and distillation of fuel. R. DRAWE (G.P. 425,316, 15.11.21).—The drying and distillation take place in a shaft divided by means of roof-shaped, transverse plates which form channels through which the heating gases pass in such a way that they do not penetrate the material but simply sweep over it, and the gases and vapours produced are led away directly, practically free from dust.

A. COULTHARD.

Production of rich gas by the aid of water-gas. O. MISCH (G.P. 425,945, 3.1.25).—The fuel heated to the limit of caking by heated water-gas passed through the coking retort, is directly transferred to the water-gas generator and also to a gas generator which supplies the gaseous fuel necessary for the heat required in the process as well as in the works, also for a gas engine which covers the needs of the process and works with respect to electric current. The chief value of the process lies in the utilisation of the heat of production of the water-gas and the transference of the heat of the coked fuel to the water-gas generator.

A. COULTHARD.

Distillation plant for finely-divided material. H. WIEDEMANN (G.P. 426,088, 19.2.24).—A shallow circular pan, fitted with a stirrer, and provided with an airtight dome for leading off the gases, carries the material. The material is charged into, and discharged from, two neighbouring sectors as close together as possible, being carried forward tangentially from the charging to the discharging position by the arms of the stirrer. The plant is claimed to be very efficient, to take up a comparatively small space, and to be subject to low heat radiation losses.

A. COULTHARD.

Distillation of caking coal. A.-G. FÜR BRENNSTOFFVERGASUNG, Assees. of O. HUBMANN (G.P. 426,690, 1.11.21).—Caking coal is preheated to about 300° before it is charged into a retort, and before it reaches the distillation zone in the retort it passes through an intermediate zone in which it is heated sufficiently to destroy the caking constituents.

L. A. COLES.

Production of gas of low moisture content from fuel of high moisture content. F. JAHNS (G.P. 427,115, 26.1.24).—After re-charging the retorts, the last retort of the series is shut off from the remainder and used as a drying chamber until steam and other gases are completely expelled, when it is again linked up in the series.

L. A. COLES.

Gaseous fuel. J. HARRIS (U.S.P. 1,584,291, 11.5.26. Appl., 6.12.23).—A mixture of $2\frac{1}{2}$—5 volumes of ether vapour with $97\frac{1}{2}$—95 volumes of water-gas is used as a fuel for cutting metals.

S. PEXTON.

Carburetted water-gas plant. G. W. SMITH and F. L. WEISSER (U.S.P. 1,585,429, 18.5.26. Appl., 9.6.20).—The plant comprises a water-gas generator together with two chequered chambers which alternately act as carburettor and superheater respectively. During each "blow" period the carbon deposit in one or other chequered chamber is burnt off by blowing air into it.

S. PEXTON.

Purification of gases. S. COULIER (E.P. 250,837, 8.10.25).—Hydrogen sulphide and hydrocyanic acid are removed from gases by washing with an alkaline solution, *e.g.*, of sodium carbonate, which may contain an iron compound or may be subsequently treated with a paste of ferrous hydroxide and water. The ferrous sulphide and excess ferrous hydroxide are removed and the decanted liquid is mixed with ferric carbonate or other iron compound in the presence of carbon dioxide; the ferric ferrocyanide is removed as a blue sludge. The filtrates and decanted liquids pass down a tower through which air is blown to convert the sodium bicarbonate into carbonate, which is used again for washing the gas. The bicarbonate solution may be regenerated by neutralising with ferrous or ferric hydroxide. Modifications and alternative methods of working the process are given.

A. C. MONKHOUSE.

Removal of dust from the gases produced in low-temperature carbonisation. DEUTSCHE LUFTFILTER-BAUGES. M.B.H. (G.P. 426,284, 13.3.23).—Mechanical filters interposed between the distillation apparatus and the condenser are kept at such a reduced temperature that a part of the distillate

passing through them is condensed. The filters are thus kept continuously and automatically moist and are able to remove all the dust completely. The material collecting in the filter agglomerates into balls and is returned automatically or otherwise to the distillation apparatus. Losses of tar and coating of the heating surfaces in the distillation apparatus with dust are avoided. A. COULTHARD.

Automatic gas analyser and control. L. YOUNG and G. W. RUGGLES (U.S.P. 1,578,697, 30.3.26. Appl., 15.6.22).—A constant-head water aspirator draws a sample of the gas through a bubbler and then into the absorption tube which is fixed in the sealed end of a two-chambered vessel containing the absorbing liquid, the second chamber being open to the atmosphere. The suction draws a quantity of the reagent up into the absorbing tube, the height to which it ascends being governed by the suction, which is constant, and the amount of carbon dioxide or other gaseous constituent which it absorbs. The spent absorbing fluid is carried back to the sealed end of the two-chambered vessel by a side tube. The height of the liquid in the absorbing tube determines the height of liquid in the gauge, which is connected up between the aspirator and the trap which follows the absorbing tube. E. S. KREIS.

Extraction of wax from lignite. A. MAILHE (F.P. 598,502, 2.8.24).—Finely powdered lignite is subjected to the action of vapours of organic solvents, such as aniline, mono- and di-methylaniline, toluidine, methyltoluidine, xylidine, methyl- and ethyl-xylidine, aniline oil, pyridine, quinoline, phenol, cresol and mixtures of these, or it is continuously stirred with the solvents themselves.
A. COULTHARD.

Motor fuel. Y. NIKAIDO (U.S.P. 1,582,420, 27.4.26. Appl., 9.7.25).—A mixture of hydrocarbons with a small proportion of a dialkyl ether of b.p. below 104° to accelerate ignition. S. BOWMAN.

Bolted steel tube still for distilling oil. J. M. SIMS and O. M. BROOKS (U.S.P. 1,581,879, 20.4.26. Appl., 24.10.24).—The still consists of a heating coil formed of bolted steel tubes lined with clay material, and a continuous coil formed from a series of tubes extending across the heating coil. The bends of the continuous coil project through the walls of the heating coil and are removable. S. BOWMAN.

Recovering gasoline. F. E. HOSMER (U.S.P. 1,581,212, 20.4.26. Appl., 14.5.20).—A mixture of permanent gas and condensable vapour is enriched with a further quantity of condensable vapour, compressed, and cooled. By evaporation of a portion of the condensed liquid the inlet gases are cooled by heat transfer, the evaporated vapours being returned to the compressor with the fresh gases.
S. BOWMAN.

Distillation of crude petroleum. E. M. CLARK, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,582,123, 27.4.26. Appl., 3.7.19).—Steam is injected during the distillation in order to give the maximum quantity of distillate at a still temperature below that at which cracking is imminent. S. BOWMAN.

Apparatus for treating hydrocarbon oils. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,582,585, 27.4.26. Appl., 11.10.19. Renewed 15.7.25).—A series of continuous stills is arranedg at different heights and fired independently. Each still is fitted with a refluxing and condensing column and so connected with the others that the charging stock can be run through any one reflux column and be preheated before entering any still. All by-pass communications are controlled by valves, thus maintaining a pressure on the oil during distillation and condensation. S. BOWMAN.

Apparatus for refining [petroleum] oils. D. PYZEL, Assr. to SIMPLEX REFINING Co. (U.S.P. 1,583,746, 4.5.26. Appl., 26.10.21).—Cold oil is delivered to the top of a tight shell containing means for spreading and dividing the oil, and is passed from the shell to a still, the vapours from which pass upward through the shell in intimate contact with the oil, which condenses and absorbs a part of them. A portion of the oil is withdrawn from the shell, cooled, and returned to the top of the shell.
H. MOORE.

Production of low-boiling hydrocarbons from distillation gases. GELSENKIRCHENER BERGWERKS-A.-G., ABTEIL. SCHALKE, and F. CASPARI (G.P. 424,724, 8.10.24).—The gases produced in the dry distillation of fuel are subjected to a cracking process outside the distillation vessel by means of the heating gases used in the distillation plant. Pure cracked products are obtained on an economic scale.
A. COULTHARD.

Refining of mineral oils. A. RIEBECK'SCHE MONTANWEKRE A.-G. (G.P. 426,157, 29.1.25. Addn. to 421,858; B., 1926, 263).—The oils are treated with titanium halides either in a finely divided or a diluted form. The halides may be precipitated on finely-divided solid carriers or dissolved in organic liquids which take no part in the reaction.
A. COULTHARD.

Purification of hydrocarbons. A. RIEBECK'SCHE MONTANWERKE A.-G. (G.P. 427,285, 22.3.25. Addn. to 421,858; B., 1926, 263).—Hydrocarbon mixtures such as mineral oil, are purified by treatment with antimony halides, in finely divided form or in solution, instead of with stannic or titanium halides.
L. A. COLES.

Production of mineral oil products free from water. F. HANSGIRG (Austr. P. 97,258, 5.10.16. Addn. to 90,467; cf. U.S.P. 1,398,856; B., 1922, 43 A).—Oils free from acids and phenols, which are light in colour, even when of high viscosity, and which need no further purification with sulphuric acid and sodium hydroxide, are obtained by distillation in the presence of substances which remove water, employing the electrical heating method of the chief patent (*loc. cit.*). A. COULTHARD.

Purifying and decolorising agent for oils. H. L. Kauffman, Assr. to Producers and Refiners Corp. (U.S.P. 1,579,326, 6.4.26. Appl., 27.5.24).—Finely-divided leverrierite is heated for 7 hrs. at 93° with 20% sulphuric acid and then thoroughly washed with water. The product is of value as a purifying, deacidifying, and decolorising agent for oils, especially lubricating oils. T. S. Wheeler.

Preparing "castor" machine oils. L. L. Rebber, Assr. to Union Oil Co. of California (U.S.P. 1,582,227, 27.4.26. Appl., 9.5.22; cf. U.S.P. 1,582,258, *infra*).—Waste alkaline liquids from the purification of petroleum lubricating oil distillates are treated with sodium chloride when a mixture of alkali naphthenates and naphthenic acids separates. It is saponified with strong caustic soda solution, and the sodium naphthenate is boiled with an excess of a solution of a soluble aluminium salt, such as aluminium sulphate, when the corresponding aluminium naphthenate separates as an insoluble rubber-like mass. This is washed, dried, and dissolved in a mineral oil, such as kerosene, to form a machine lubricating oil which retains its viscosity for long periods and at high temperatures. It clings to both wet and dry surfaces. T. S. Wheeler.

Castor machine oils. De R. Frizell, Assr. to Union Oil Co. of California (U.S.P. 1,582,258, 27.4.26. Appl., 9.5.22).—The viscosity and the stability of lubricating oils, including those prepared as described in U.S.P. 1,582,227 (*supra*), are increased by the addition of up to 10% by volume of sodium naphthenate. The addition of less than 1% of water to the aluminium naphthenate increases its solubility in the mineral oil. T. S. Wheeler.

Treating lubricating oils. E. G. Acheson (U.S.P. 1,548,588, 11.5.26. Appl., 19.3.21).—Mineral lubricating oils are extracted with a suitable mixed solvent, such as an equal weight of 50% aqueous acetone, when about 80% of the oil with a higher lubricating power than the original remains undissolved and is separated. The acetone is distilled from the extract and the extracted oil is separated from the aqueous residue. T. S. Wheeler.

Preparing soluble oils. De R. Frizell and B. A. Stagner, Assrs. to Union Oil Co. of California (U.S.P. 1,582,257, 27.4.26. Appl., 1.4.22).—Waste alkaline liquids from the purification of petroleum distillates are acidified and the naphthenic acids which separate are saponified with strong caustic soda solution. The sodium naphthenate is dried, mixed with about one-fourth of its volume of cresol, and dissolved in a mineral oil such as kerosene. The product forms stable emulsions with water which can be used as sprays for fruit trees etc., as sheep dips, as substitute for Turkey-red oil, lubricants, etc. It does not corrode steel or brass. T. S. Wheeler.

Removing wax from [petroleum] oil. H. F. Glair and O. E. Bransky, Assrs. to Standard Oil Co. (U.S.P. 1,582,923, 4.5.26. Appl., 7.7.22).—Wax is separated from lighter substantially uncracked distillates by admixing a hydrocarbon oil of lower viscosity and heavier than burning oil, chilling and filter-pressing the mixture. H. Moore.

Recovery of metallic halides from hydrocarbon sludges. P. S. Danner, Assr. to Standard Oil Co. (U.S.P. 1,582,131, 27.4.26. Appl., 19.5.25).—The sludge produced during the conversion of hydrocarbons in the presence of halides is subjected to simultaneous cracking and hydrogenation, whereby lighter saturated hydrocarbons are formed and the halide is liberated for further use. S. Bowman.

Gilsonitic products. C. N. Forrest, H. P. Hayden, and O. R. Douthett (E.P. 252,051, 30.1.25).—See U.S.P. 1,459,328 and 1,524,859; B., 1923, 879 A; 1925, 276.

Feeding or/and discharging of retorts for distillation or other purposes. A. L. J. Queneau (E.P. 252,268, 24.2.25).

Automatically cleaning the interior walls and moving parts and adjuncts of retorts while in operation. A. L. J. Queneau (E.P. 252,269, 24.2.25).

Oil-gas generators. J. Zwicky (E.P. 252,080, 19.12.24 and 17.10.25).

Separating hydrogen from water-gas, coke-oven gas, etc. (F.P. 601,774).—See VII.

III.—TAR AND TAR PRODUCTS.

Elimination of sulphur impurities from technical cresols and petroleums. G. L. Stadnikov, N. M. Gavrilov, and V. E. Rakovskii (J. Chem. Ind. [Moscow], 1925, 2, 315—319; Chem. Abstr., 1926, 20, 1706).—The speed of condensation of thiocresols with aldehydes is much greater than that of cresols. A condensing agent (1% of petroleum sulphonic acids) and an aldehyde (3% of dextrose or formaldehyde solution) are mixed with an equal quantity of the oil, and the mixture is added in small portions to the bulk of the oil in a distilling flask, with stirrer and condenser, and heated at 120°. The water gradually distils off; the oil is then distilled below 140° under reduced pressure. The sulphur content of a crude coal-tar cresol was thus reduced from 2·24% to 0·01%. When more than 10—15% of neutral oils is present, sulphur is not normally removable by this method. A. A. Eldridge.

See also A., June, 578, **Distillation of heterogeneous ternary mixtures. I. System water-benzene-toluene** (Barbaudy). 604, **Hydrolytic decomposition of sulphonic acids of isomeric xylenes in relation to purification of commercial xylene** (Kishner and Vendelstein).

Low-temperature tar oils for Diesel engines. Spilker.—See II.

Patents.

Manufacture of hard pitch. Prodor Fabr. de Prod. Organiques S.A., and M. Lévy (F.P. 596,331,

23.12.24).—Tar or bituminous material is distilled as rapidly as possible under a high vacuum, and superheated steam is passed into the mass towards the end of the process. The crude material is treated with solvents to precipitate free carbon. L. A. COLES.

Preparation of hydrocarbons or lower phenols from low-temperature tar phenols or tar oils containing them. OBERSCHLESISCHE KOKSWERKE & CHEM. FABR. A.-G., and A. SUPAN (G.P. 422,035, 16.7.21).—The material is distilled with steam at high temperatures over such substances as iron or coke which liberate hydrogen from steam. The products formed are chiefly benzene and its derivatives. If low-boiling benzol is required, high temperatures are employed; with low temperatures the higher-boiling derivatives are formed. Phenol itself is obtained in good yield if the conditions are arranged for an incomplete decomposition. Low-temperature tar phenols distilled with superheated steam over iron turnings at 650° yield 60—65% of neutral oils (hydrocarbons); the phenols, mainly phenol, amount to about 7% of the distillate. A. COULTHARD.

Purification of crude benzol. OBERSCHLESISCHE KOKSWERKE & CHEM. FABR. A.-G., and F. RUSSIG (G.P. 423,787, 4.12.20).—The constituents which are the cause of a bad sulphuric acid reaction are removed by fractional distillation with a good condensing column, after which the main part of the benzol is distilled off, with or without a column. In order to avoid the presence of runnings having a bad sulphuric acid reaction, the fractionation is interrupted before the heating coil becomes exposed, and the still either refilled with crude benzol and the process repeated, or a number of residues are collected, and a fraction consisting of benzene and its nearest homologues and having a good sulphuric acid reaction is distilled off until the temperature of the residue rises to about 145°. A. COULTHARD.

Production of light oils from low-temperature tar or heavy oils. ZECHE M. STINNES (G.P. 426,111, 12.8.21).—Low-temperature tar or its distillation products are subjected to superheated steam (insufficient in amount to produce an effect similar to a vacuum) at comparatively low temperatures (600—700°), with or without the use of gases or substances which offer as large a surface as possible to the reaction mixture. A. COULTHARD.

IV.—DYESTUFFS AND INTERMEDIATES.

Reduction of nitro-derivatives by means of sodium amalgam. G. PELLEGRINI (Giorn. Chim. Ind. Appl., 1926, 8, 175—178).—Reduction of nitrobenzene by means of sodium amalgam yields hydrazobenzene, but under similar conditions *o*-nitrotoluene and *o*-nitroanisole give almost exclusively the corresponding mono-amines. In presence of alcohol, however, the reduction of these compounds gives hydrazotoluene and hydrazo-anisole in good yields. T. H. POPE.

See also A., June, 595, **Oxidation of organic substances by copper oxide** (STEOPOE). 606, **Minimal concentration of acid for sulphonation** (COURTOT and BONNET). 607, **Benzidine derivaderivatives of thiocarbamide** (PINTO). 610, **Thymolsulphonephthalein** (ORNDORFF and CORNWELL). 618, **Colouring matters of sandalwood. Constitution of santalin** (DIETERLE and STEGEMANN).

PATENTS.

Manufacture of dyestuffs [containing zinc]. SOC. CHEM. IND. IN BASLE (E.P. 241,572, 15.10.25. Conv., 16.10.24).—Very fast dyes, capable of chroming, containing zinc, and possessing good solubility and equalising power are obtained by treating azo dyes produced by the action of reducing agents on the products obtained by coupling nitrated diazotised 1-amino-β-naphthol-4-sulphonic acid and a naphthol (cf. U.S.P. 1,521,206; B., 1925, 165), with reagents that yield zinc, such as zinc chloride or hydroxide. For example, an aqueous solution of 43·2 pts. of the reduced dye from nitrated diazotised 1-amino-β-naphthol-4-sulphonic acid and α-naphthol is stirred for some time, at ordinary temperature, with a solution of zinc chloride and potassium hydroxide. After neutralising the excess of alkali, the *zinc* compound of the dye is salted out as a blackish-brown powder which gives a violet solution in water, bluish red on adding caustic alkali. The dye gives violet to brownish shades on wool which become grey or black when after-chromed. A. COULTHARD.

Manufacture of [benzanthrone] dyestuffs and intermediates. R. F. THOMPSON, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 251,313, 21.10 and 12.11.24).—Benzanthrone, when treated with manganese dioxide and sulphuric acid is converted into a mixture of a dibenzanthronyl differing from the known one, and a hydroxybenzanthrone capable of alkylation and giving a dye when fused with alkali hydroxide (cf. E.P. 218,255, B., 1925, 538). The mixture is separated by means of alkali hydroxide solution (in which the hydroxybenzanthrone is soluble). The dibenzanthronyl, brown needles, m.p. above 300°, is converted by treatment with alkali and alcohol into dibenzanthrone of high purity. A. COULTHARD.

Production of vat [dibenzanthrone] dyestuff. D. G. ROGERS and L. C. DANIELS, Assrs. to NATIONAL ANILINE & CHEM. CO. (U.S.P. 1,583,258, 4.5.26. Appl., 2.7.21).—100 pts. of potassium hydroxide and 420 pts. of a mineral oil with a boiling point a little above 230° are heated to 215—230° and 24 pts. of dextrin or other reducing agent, such as starch or an aminophenol, mixed with 100 pts. of sublimed benzanthrone are added. The temperature is maintained at 215—230° until reaction is complete, when the mixture is cooled, the solid matter is suspended in water, and dibenzanthrone formed by passing air through the boiling suspension. The presence of the inert liquid boiling at about the optimum temperature of the reaction enables accurate temperature control to be achieved and protects the melt from the action of air. T. S. WHEELER.

Manufacture of grey to black vat [dibenzanthrone] dyes. BADISCHE ANILIN- & SODA-FABR., Assees. of P. NAWIASKY and E. KRAUCH (G.P., 423,878, 29.6.24; Addn. to G.P. 411,693, cf. E.P. 232,799; B., 1925, 538).—The sulphurisation of the nitration products of dibenzanthrone is carried out in the presence of solution or suspension media. The dyes are the same as those of the chief patent.
A. COULTHARD.

Manufacture of thioindigo derivatives. H. DODD, W. C. SPRENT, and THE UNITED ALKALI CO., LTD. (E.P. 251,321, 29.12.24).—A dichlorobenzene is treated with chlorosulphonic acid and the dichlorobenzenesulphonyl chloride is reduced, *e.g.*, by means of hydrochloric acid and zinc or iron, to dichlorothiophenol, which is separated by distillation. This is treated with chloroacetic acid and aqueous caustic alkali. The dichlorophenylglycollic acid obtained by acidification, is converted into tetrachlorothioindigo by the action of sulphuric acid.
A. COULTHARD.

Manufacture of basic dyes of the Malachite Green series. A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 251,511, 23.9.25).—Basic *dyes* showing purity of shade, good equalising power, and great affinity for artificial silk are obtained by condensing a 2 : 3 : 5-trichloro- or 2 : 3 : 4 : 5-tetrachlorobenzaldehyde with two mols. of an *N*-mono- or dialkyl-derivative of an arylamine, or a mixture of these, and oxidising the leuco-base produced. 2 : 3 : 5-*Trichlorobenzaldehyde*, m.p. 75—76°, is obtained from 2 : 3 : 5-trichlorotoluene. 2 : 3 : 4 : 5-Tetrachlorobenzaldehyde, m.p. 106—106·5°, is made by passing chlorine through a solution of 4-chloroacet-*o*-toluidide, hydrolysing the 3 : 4 : 5-trichloroacet-*o*-toluidide (the main product), and replacing the amino-group by chlorine by the Sandmeyer reaction; chlorination of the side chain results in 2 : 3 : 4 : 5-tetrachlorobenzylidene chloride which on hydrolysis gives the aldehyde. A method of obtaining the 2 : 3 : 4 : 5-tetrachloroaldehyde mixed with the 2 : 3 : 4 : 6-compound from mixtures of 3 : 4 : 5- and 3 : 5 : 6-trichloroacidyl-*o*-toluidines (the starting point for these being *o*-nitrotoluene) is also given. The two tetrachloro-aldehydes can be separated by making use of the different solubilities of their bisulphite compounds. The dye from 2 : 3 : 5-trichlorobenzaldehyde and ethyl-*o*-toluidine gives blue shades, whilst the *dye* from 2 : 3 : 4 : 5-tetrachlorobenzaldehyde and diethylaniline gives bluish-green shades. The dyes can be used for the manufacture of lakes and pigments. A. COULTHARD.

Triphenylmethane dye. W. DUISBERG, W. HENTRICH, and W. SCHEPSS, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,582,909, 4.5.26. Appl., 30.6.24; cf. E.P. 12,130 of 1910 and 12,378 of 1912 and U.S.P. 1,244,149; B., 1910, 1297 A; 1912, 809 A; 1918, 5 A).—Triphenylmethane compounds containing a substituent such as Cl, NO_2, SO_3H, CH_3O etc. in the *p*-position to the methane carbon are condensed with halogenated aromatic bases containing a substituent in the *o*-position, such as 2-chloro-4-toluidine or the 2 : 5-, 2 : 6-, or 3 : 4-isomers, or 4 : 6-dichloro-2-toluidine, or 4-chloro-2-anisidine, to yield products which dye wool bluish-red to reddish-violet shades, reddish-violet on after-chroming and fast to milling and potting. These compounds can be rendered soluble in water and hence more suitable for printing by sulphonation with a mixture of oleum and sulphuric acid. T. S. WHEELER.

Making [anthraquinone] dyes. J. B. MARVIN, JUN. (U.S.P. 1,580,265, 13.4.26. Appl., 5.2.21).—Sodium 1 : 4-dichloroanthraquinone-5-sulphonate is heated under pressure at 200° for 10 hrs. with *p*-toluidine-*o*-sulphonic acid in presence of water and of sufficient calcium carbonate to keep the mixture neutral. The reaction is carried out in presence of about 1% of silver or mercury or of an amalgam of these metals (a silver-lined autoclave may be employed). The product is dissolved in water and salted out hot. The yield of green dyestuff is greater and the quality is better than is obtained by the usual method of preparing this product (cf. F.P. 384,471, B., 1908, 442). T. S. WHEELER.

Producing *N*-dihydro-1 : 2 : 1′ : 2′-anthraquinoneazine and derivatives. M. S. THOMPSON, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,580,700, 13.4.26. Appl., 9.6.22).—A melt of potassium hydroxide (30 pts.) and sodium hydroxide (15 pts.) is treated at 210° with sodium ethoxide (5 pts.) and potassium chlorate (1·75 pts.), and finally with 12 pts. of 2-aminoanthraquinone. After 30 min. the mixture is poured into vats when a satisfactory yield of *N*-dihydro-1 : 2 : 1′ : 2′-anthraquinoneazine is obtained. Other alkoxides or phenoxides may be be employed, sodium butoxide and sodium phenoxide being particularly effective. T. S. WHEELER.

Anthracene dye. R. J. GOODRICH, Assr. to NEWPORT CO. (U.S.P. 1,581,111, 20.4.26. Appl., 4.4.25).—The condensation product of 2 : 2′-dimethyl-1 : 1′-dianthraquinonyl is dissolved in fuming sulphuric acid and brominated in the presence of a catalyst (*e.g.*, iodine). The mixture is poured into water, filtered, and washed. The paste obtained may be used directly for dyeing in a hyposulphite vat, and produces orange-red shades on cotton.
R. B. CLARKE.

Vat dyes of the naphthanthraquinone series. BADISCHE ANILIN- & SODA-FABR. (F.P. 599,038, 30.5.25. Conv., 4.7 and 17.12.24).—Naphthanthraquinone or a derivative is heated with anhydrous aluminium chloride at temperatures below 250° in the presence or absence of anhydrous halides of other metals such as sodium chloride or ferric chloride. With solvents such as phthalic anhydride the dye is formed at 170°. Cotton is dyed from the vat in very fast blue to violet shades. The dye obtained from chloronaphthanthraquinone (obtained by condensing *β*-chlorophthalic acid with naphthalene) dyes cotton violet-blue.
A. COULTHARD.

Brown copper-containing aromatic amine-sulphur dyes and process of making them. J. FLACHSLAENDER and L. J. BURGESS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,580,716, 13.4.26. Appl., 19.5.24).—The product obtained by heating an aromatic amine and sulphur is dissolved in sodium hydroxide or sodium sulphide solution, copper sulphate or chloride is added, and the mixture evaporated to dryness and baked at about 300°. The products dye cotton fast brown to khaki shades. The dyestuff from *m*-toluylenediamine gives on unmordanted cotton full cutch shades fast to washing, milling, and acids. The colours obtained vary somewhat with the temperature of the final baking. T. S. WHEELER.

Purification of phenolphthalein. J. G. SCHUDEL, Assr. to NATIONAL ANILINE & CHEM. Co. (U.S.P. 1,583,372, 4.5.26. Appl., 5.2.23).—250 pts. of crude phenolphthalein are dissolved in 425 pts. of 15% caustic soda solution and filtered, and the filtrate is added to 160 pts. of iced 50% sulphuric acid, or alternatively is treated with 100 pts. of denatured alcohol and sufficient 50% sulphuric acid to give a distinct acidity. 235 pts. of the precipitated phenolphthalein are dissolved in 1415 pts. of denatured alcohol, hydrochloric acid is added to give a faint acidity, and the solution is boiled under reflux for 15 hrs. with 20 pts. of activated charcoal and 3·6 pts. of zinc dust, then cooled, and filtered. On concentration and cooling practically pure phenolphthalein separates. T. S. WHEELER.

Producing naphtholsulphonic acids. P. H. FALL and I. E. LEE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,580,714, 13.4.26. Appl., 30.6.20).—α-Naphthylamine-4- or -5-sulphonic acid is converted into the sulphurous ester of α-naphtholsulphonic acid by treatment with sodium hydrogen sulphite solution ; and the product is boiled in aqueous solution with an alkaline-earth or other hydroxide which yields a sparingly soluble sulphite. Hydrolysis is complete in 3 hrs. and the yield of the naphtholsulphonic acid is almost theoretical. T. S. WHEELER.

Purification of α-nitronaphthalene. H. F. WHITTAKER and W. WOLLASTON, Assrs. to E. I. DU PONT DE NEMOURS AND Co. (U.S.P. 1,581,258, 20.4.26. Appl., 1.7.20).—Crude α-nitronaphthalene mixed with 10% of its weight of solvent naphtha is heated at 50—55° until a homogeneous solution is obtained. When this is cooled at 25° with agitation, minute crystals are formed, which are separated by centrifuging. The product has m.p. not lower than 54·4°. R. B. CLARKE.

Purification of anthraquinone. H. F. LEWIS, Assr. to NATIONAL ANILINE & CHEMICAL Co. (U.S.P. 1,583,297, 4.5.26. Appl., 7.1.21).—100 pts. of crude anthraquinone are mixed with 1200 pts. of 5% caustic soda and reduced to anthranol by addition of 75 pts. of zinc dust, the temperature being maintained at 30°. The mixture is filtered under steam pressure, and the filtrate is blown with air to precipitate anthraquinone, which is filtered off and washed with alkali and water. T. S. WHEELER.

Purification and isolation of anthraquinone-β-sulphonic acid. I. GUBELMANN and R. J. GOODRICH, Assrs. to NEWPORT Co. (U.S.P. 1,584,372, 11.5.26. Appl., 9.5.25).—The product obtained by heating 1 pt. of anthraquinone with 1 pt. of sulphuric acid containing 40% SO_3 at 145° for 2 hrs. is poured into 10 pts. of cold water and filtered, and the filtrate is concentrated to d^{25} 1·067 and treated with concentrated ammonia until slightly alkaline, heated to 90° for 1 hr., and cooled to 25°. The pure ammonium salt of anthraquinone-β-sulphonic acid separates and is filtered off after 12 hrs. The addition of soluble ammonium salts further decreases its solubility. T. S. WHEELER.

Diazotisation of organic compounds. C. P. HARRIS and N. M. ELIAS (U.S.P. 1,585,145, 18.5.26. Appl., 17.6.24).—An excess of aniline or other diazotisable amine is mixed with 93% sulphuric acid to yield a paste which can be agitated. If preferred the excess of aniline which is used as a diluent may be replaced by an inert liquid. About 2% of hydrochloric or other acid, the aniline salt of which is soluble in the diluent, is added as a catalyst and then solid sodium nitrite is slowly added at 45°. The sodium sulphate which separates removes all water present and the diazobenzene formed is separated or treated in any desired manner. T. S. WHEELER.

Black ink [from nitro-dinaphthylene dioxide]. (F.P. 600,390).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Absorption of moisture by various fibres under different atmospheric conditions. H. FAY (ZELLSTOFF U. PAPIER, 1926, 6, 201—203).—Different types of paper-making fibres react towards atmospheric moisture at different rates and to different equilibria. Comparative observations were made by teasing out the fibres of the various half-stuffs, drying them completely, exposing them to atmospheres of various known relative humidities for 12 hrs., and determining the moisture taken up. Mitscherlich sulphite pulp absorbed, under similar conditions, about 1% more moisture than Ritter-Kellner pulp, and unbleached pulps of each type absorbed slightly more than the same pulps bleached. The absorption of moisture by the fibres did not rise proportionally with the degree of saturation of the atmosphere. The absorption of moisture by straw cellulose was almost identical with that of unbleached Mitscherlich wood pulp, but aspen cellulose, while behaving in the same way in atmospheres of low humidity, showed a remarkable increase when exposed to atmospheres above 70% relative humidity, a value of 13·9% being recorded on exposure to air at 80% saturation. The absorption of moisture by bleached linen showed a regular rise

with increase of humidity but in no case was it so high as that of wood pulp under the same conditions, *e.g.*, bleached wood pulp 10·7; bleached linen 9·6% in air at 74% saturation. With esparto pulp the moisture was absorbed extremely slowly in atmospheres of low humidity (up to 50% saturation) but became equivalent to that of wood pulp in an atmosphere of 80% saturation. The variable response of the different fibres to atmospheric changes is reflected in the cockling of the paper in the printing shop, and the practice of separating and exposing the sheets by hanging them up in the shop for some hours before printing is undertaken is an important precaution. J. F. Briggs.

Distinguishing viscose and cuprammonium silks by colour reactions. P. Krais (Papier-Fabr., 1926, 24, 330—331).—The best colour reactions are that of Rhodes, in which the silk is boiled with a solution of silver nitrate, sodium thiosulphate, and sodium hydroxide, and that of Cassella, in which the material is dyed with Naphthylamine Black 4 B. In Rhodes' test viscose silk is coloured a dark brownish-black, whilst cuprammonium silk becomes only a very light grey. In Cassella's test viscose silk is dyed a light reddish-grey and cuprammonium silk a dark bluish-grey; viscose silk from linters is dyed a light bluish-grey. It is recommended to apply both tests in doubtful cases. A. Geake.

Guignet cellulose from wood cellulose and wood. C. G. Schwalbe and W. Lange (Z. angew. Chem., 1926, 39, 606—608).—Cellulose dissolves completely in 62% sulphuric acid to a colloidal solution, from which it may be reprecipitated by the addition of electrolytes, or by dilution with water, alcohol, or ether. The Guignet cellulose prepared by dissolving 1 g. of sulphite-cellulose in 7 c.c. of 62·53% sulphuric acid and precipitating after 5 hrs. by the addition of water, is completely redissolved by water to a milky solution, and is dissolved to the extent of 95% by 10—17·5% sodium hydroxide solution. The yield is 95%. The cellulose, copper, and hydrolysis numbers are 0·4, 8·2, and 14·7, compared with 0·2, 2·4, and 5·9, respectively, for the original sulphite-cellulose. (The cellulose number is a measure of the degree of "swelling"; it is determined by immersing the sample in Fehling's solution and, after thorough washing, ascertaining the quantity of copper fixed.) By twice dissolving in 10% sodium hydroxide and reprecipitating with acid a yield of 30% of a substance having a copper number of only 2·6 is obtained. The pentosan content of the Guignet cellulose is 0·56%, compared with 5·2% for the sulphite-cellulose, indicating that pentosan is more readily hydrolysed by sulphuric acid than is cellulose. By this process the cellulose is readily dissolved from spruce wood, leaving 29—30% of lignin; the Guignet cellulose obtained contains only 1·4% of pentosan. A. Geake.

Alkali-soluble cellulose. T. Lieser (Cellulosechem., 1926, 7, 85—88).—When cellulose is dissolved in a saturated aqueous solution of hydrochloric acid at –5°, containing 46·5% HCl, and reprecipitated after a few minutes by dilution, a 99% yield is obtained of a substance which, if not dried, is completely soluble in 8% sodium hydroxide solution. By such treatment the copper number (Schwalbe) is increased from 0·09 to about 0·6. If the product is dried 8% sodium hydroxide causes swelling but not dissolution. By more prolonged treatment with acid, *e.g.*, for 4 hrs. at 0°, the yield obtained by dilution is reduced, and the product is completely soluble in 8% sodium hydroxide even after drying; the copper number is increased to 2·2. Alkali-soluble cellulose has all the reactions of normal cellulose. It is more easily etherified and esterified, but acetolysis is more difficult than with normal cellulose; viscose films or threads prepared from it are unstable. The solution in 8% sodium hydroxide undergoes no change when kept, and the cellulose is precipitated by heating, diluting, or by adding alcohol. The solubility is probably due to the formation of a molecular compound which, unlike that of the original cellulose, is soluble. The insolubility after drying may be due to the great reduction in surface area. A. Geake.

Determination of α-cellulose. H. Bubeck (Papier-Fabr., 1926, 24, Fest- u. Ausland-Heft, 66—71).—When cellulose is mercerised with 17·5% sodium hydroxide, filtered off, and washed, the sum of the hemicellulose in the filtrate, as determined with chromic acid, and of the undissolved α-cellulose is less than 100%. This is due to the more dilute alkali formed during washing dissolving material which is not dissolved by the mercerising alkali. A higher extract of hemicellulose is therefore obtained if the mercerising alkali is diluted before filtering, and the maximum value is obtained when the diluted solution contains 9 g. of sodium hydroxide per 100 c.c. The amount extracted depends, to a small extent, on the duration of mercerisation, increasing by about 0·2% between 0·5 and 1·5 hrs. It is more dependent on the temperature, and diminishes by about 2% when the temperature is raised from 12° to 27°. To determine α-cellulose 3 g. of the material are mercerised at 18° with 20 c.c. of 17·5 wt.-% sodium hydroxide. After ½ hr. 80 c.c. of 5—6 vol.-% sodium hydroxide are added, and the residue after filtration is washed with 50 c.c. of 8—9 vol.-% sodium hydroxide, and then with water, acetic acid, and water. It is then dried and weighed. Hemicellulose may be determined in an aliquot portion of the filtrate by oxidation with chromic acid. It is proposed to re-define α-cellulose in accordance with this method of determination. A. Geake.

Colloidal properties of nitrocellulose sols in mixed solvents. A. Highfield (Trans. Faraday Soc., 1926, 21, 57—81).—See B., 1926, 188.

See also A., June, 573, **Adsorption of alkali by cellulose** (Liepatov). 576, **Cellulose dispergation in concentrated aqueous solutions of strontium thiocyanate, bromide, and chloride** (Von Weimarn and Aoki); **Cellulose dispergation in concentrated solutions of barium thiocyanate and bromide** (Von Weimarn and Kataoka);

Cellulose dispergation in concentrated solutions of calcium bromide and chloride (Von Weimarn and Otsuka); **Ability of thiocyanates and halides of alkaline-earth metals to produce dispergation of cellulose** (Von Weimarn); **Cellulose dispergation in aqueous sodium citrate and calcium chloride solutions of extremely low concentrations** (Von Weimarn and Hori).

Patents.

Manufacture of artificial silk and of artificial threads or filaments. J. E. G. Lahousse, Assr. to Soc. Fabr. Soie "Rhodiaseta" (U.S.P. 1,583,475, 4.5.26. Appl., 26.9.24).—A solution of a cellulose derivative in a volatile solvent is spun by the "dry" method in a closed cell by introducing into the cell a gaseous evaporating medium previously richly laden with a determined proportion of vapours of the solvent, but not saturated at the temperature prevailing in the cell. By this means the cross-section of the filaments produced is subject to control.
J. F. Briggs.

Production of artificial silk from cellulose xanthate. H. Lumière (F.P. 602,711, 13.12.24).—Sodium bicarbonate solution, with or without the addition of neutral salts such as sodium, ammonium, or magnesium sulphate, is used for precipitating cellulose from cellulose xanthate solutions.
L. A. Coles.

Manufacture of celluloid-like masses from the esters or ethers of cellulose. A. Romahn (E.P. 238,253, 10.8.25. Conv., 9.8.24).—An ester or ether of cellulose, together with a non-volatile gelatinising agent, is dissolved in a volatile solvent and dried rapidly on hot rolls; the plastic mass may be at once worked up into sheets or other articles. The use of a solvent in conjunction with a non-volatile gelatinising agent enables the liquid to be filtered and to be subsequently dried in a short time. Substances with a glass-like transparency are thus rapidly manufactured. In an example, nitrocellulose and acetanilide are kneaded with alcohol and the mixture is dried for 20 min. at 90°.
A. Geake.

Cellulose ether film. W. R. Webb, Assr. to Eastman Kodak Co. (U.S.P. 1,583,709, 4.5.26. Appl., 20.6.25).—A film which maintains its flexibility after prolonged heating at 65° is composed of colloidised, water-insoluble ethylcellulose having distributed therein an inorganic alkali which gives the film an alkalinity in excess of p_H 7.
J. F. Briggs.

Making paper pulp. F. K. Fish, jun., Assr. to Wood Products and By-Products Corp. (U.S.P. 1,574,026, 23.2.26. Appl., 5.11.20).—Before submitting wood to the usual digestion process the complex resins and gums are rendered more soluble by treatment with circulating, superheated water which contains the volatile constituents from the wood previously extracted in a similar process. The pressure is lowered periodically to facilitate extraction of volatile substances.
R. B. Clarke.

Treating [paper] pulp. W. D. Gregor, W. M. Osborne, and A. J. Kemzura (U.S.P. 1,579,525, 6.4.26. Appl., 14.7.24).—Washed pulp prior to bleaching is treated at ordinary temperatures with dilute alkali or acid solution according as it has received an acid or alkaline boil. This treatment converts many of the impurities in the pulp into water-soluble compounds and effects about 25% reduction in the quantity of bleaching liquor required.
T. S. Wheeler.

Preparation of sulphite liquor. Lurgi Apparatebau-Ges. m.b.H. (G.P. 426,386, 8.2.25).—The gases from sulphur burners are cooled and led through an electrical precipitator of known type, to separate selenium and arsenic.
A. Coulthard.

Regenerating black liquor [from sulphate-cellulose manufacture]. A. H. White, Assr. to J. E. Alexander and E. G. Goodell (U.S.P. 1,580,269, 13.4.26. Appl., 2.4.25; cf. U.S.P. 1,197,983 and 1,374,889, B., 1916, 1106 A; 1921, 380 A).—Concentrated black liquor is heated with lime and sodium sulphate to about 650°. The sodium sulphate is rapidly reduced to sulphide, and if the product is treated with water the lime causticises the sodium carbonate also present and a solution of sodium hydroxide and sodium sulphide suitable for the manufacture of pulp is obtained. Alternatively the black liquor may be destructively distilled before lime is added. The addition of lime reduces the reduction temperature of sodium sulphate some 300°.
T. S. Wheeler.

Production of sulphur, sulphur compounds, and hydrocarbons from sulphite-cellulose waste liquor. A.-G. für Zellstoff- & Papier-Fabr., and M. Steinschneider (G.P. 427,540, 7.11.24).—The waste liquor is heated strongly in the presence of hydrogen at the ordinary or increased pressure.
L. A. Coles.

Boxes employed in spinning artificial silk. J. Brandwood, A. Stocker, and Twyver Works, Ltd. (E.P. 252,033, 15.11.24).

Bituminous impregnating compositions (E.P. 251,323).—See XIII.

Recovering by-products from plant substances (U.S.P. 1,574,614).—See XIII.

Removing nitration acids from nitrocellulose (G.P. 424,941).—See XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Printing dado effects in one or more colours. Koechlin Frères. (Sealed Note 1060, 19.11.1898. Bull. Soc. Ind. Mulhouse, 1926, 92, 161—162. Report by P. Binder, *Ibid.*, 162—163).—Fabric is printed with a dado ground (*e.g.*, ruled with fine lines) by means of a colour paste containing no mordant, then dried, overprinted with a mordant, and steamed; or these operations are carried out in the reverse order. The fabric is then washed and

soaped in the usual manner, colour being fixed only in those parts covered with the mordant. Red or pink, puce, and violet effects are obtained by means of alizarin and aluminium, chromium, and iron mordants respectively. Similar blue effects are obtained by first printing Ketone Blue and then overprinting with caustic soda of d 1·16—1·18, afterwards washing and soaping. Multicolour effects are obtained by overprinting a mordant on fabric previously printed in several colours with, for example, alizarin for pink, garnet, and violet; extract of Persian berries for yellow; Alizarin Viridine for green; Persian berry extract and Alizarin Viridine for olive etc. Black effects are obtained by means of logwood and a chromate, ferricyanide, or a salt of iron, or with a mixture of Alizarin Bordeaux, Persian berry extract, and an iron mordant. Tinted grounds are produced by addition of a suitable dye to the mordant printing paste. Binder reports favourably but draws attention to a similar process of Persoz. A. J. Hall.

[Device for] preventing the rapid decomposition of ice colours [during printing]. C. Winternitz (Sealed Note 1848, 20.7.08. Bull. Soc. Ind. Mulhouse, 1926, **92**, 163—164. Report by H. Wagner, *Ibid*, 164).—In printing ice colours during the summer months, the usual wooden furnishing rollers of the printing machines are replaced by hollow iron or copper rollers each closed at one end and having a coverable opening at the other by which the roller is filled with ice or other freezing mixture. The rollers of padding or dyeing machines containing diazo solutions may be cooled similarly. Wagner reports that a similar method of cooling yielded excellent results in one printing works.
A. J. Hall.

[Printing] coloured reserves on coloured grounds both obtained with basic dyes. A. Scheurer (Sealed Note 1999, 11.5.10. Bull. Soc. Ind. Mulhouse, 1926, **92**, 165. Report by H. Mangold, *Ibid.*, 165—166).—Fabric impregnated with tannic acid is printed with a paste containing a salt of antimony (*e.g.*, sodium antimony fluoride), antimony oxide, and a basic dye, then overprinted with another basic dye, steamed, passed through a fixing solution containing tartar emetic, washed, and soaped. Mangold reports favourably on the process.
A. J. Hall.

Printing imitation embroidery or plaited effects on woven fabrics. J. Frossard, C. Rebert, and B. Lothareff (Sealed Note, 2281, 20.10.13. Bull. Soc. Ind. Mulhouse, 1926, **92**, 167—168. Report by H. Wagner, *Ibid.*, 169—170). —Embossed imitation embroidery effects fast to soap and rubbing are obtained by printing fabric with a solution of cellulose acetate in acetic acid by means of an engraved roller, the cellulose acetate being precipitated by means of water at the time of printing. For this purpose, the engraved roller pressing against an upper rubber roller is supplied with a solution containing 140 pts. of cellulose acetate ("Sericose," Bayer) and 860 pts. of acetic acid of d 1·052 by means of an ordinary furnishing box having the necessary doctors, the fabric, before impression, being moistened by passage over a roller lapped with woollen fabric maintained wet by means of an overhead perforated steam pipe. The wet fabric precipitates the cellulose acetate on the engraved roller and at the same time the latter prints the precipitated cellulose acetate upon the fabric except from the engraved portions. Subsequently the fabric is led through a bath of boiling water whereby the printed cellulose acetate is completely fixed. Suitable dyes may be incorporated with the cellulose acetate printing solution.
A. J. Hall.

Adsorption of electrolytes by cotton yarn. Theory of mercerisation. Liepatov.—See A., June, 573.

Patents.

Colouring wood (G.P. 422,124).—See IX.

Colourless compounds containing sulphur (E.P. 242,974).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of sulphur trioxide in burner gases. E. Schmidt (Z. angew. Chem., 1926, **39**, 732—733).—The sulphur trioxide in burner gases produced under normal conditions is moist and not dry (cf. Remy, B., 1926, 255) and Gille's method of carrying out its determination (*ibid.*, 437) is criticised on this and other grounds. The wadding filter retains only 20 or 25% of the total trioxide and the suggested rate of streaming is too high. A reliable method is outlined in which 250 c.c. of gas are passed in the course of 5—8 min. through two flasks of iodine solution (25 c.c. and 5 c.c. of 0·1N-solution, respectively, made up to 100 c.c.) followed by a flask containing sodium hydroxide solution (25 c.c. of 0·1N-solution in 100 c.c.). The gas is broken up into minute bubbles on entering the solution. The sulphur dioxide is absorbed in the first flask and the trioxide in the second; if the concentration of the dilute iodine solution is doubled, most of the trioxide is absorbed in the alkali. The method enables sulphur dioxide and trioxide to be determined in the same specimen of gas. S. K. Tweedy.

Cyclic process for absorbing ammonia from coke-oven gases. L. Fokin (J. Chem. Ind. Russ., 1926, **2**, 319—329; Chem. Zentr., 1926, I., 3372).—Distilled water is circulated continuously through scrubbers for absorbing ammonia from coke-oven gases, and through a distillation column for expelling the ammonia, whence the water is returned to the scrubbers. The gas liquor is passed through the distillation column together with the solution from the scrubbers, and as the solution in the column is further diluted by the live steam used for heating it, there is an excess of water leaving the column, although some distils over with the ammonia. The excess water, which contains 0·2—0·5% of ammonia, mainly as the sulphate, is used for wetting the coal

in the ovens, so that the ammonia is not lost. The presence of ammonium salts in the liquor passing through the scrubbers does not diminish its absorptive capacity for ammonia. The hot water leaving the distillation column contains no solid particles, and is passed through heat-exchangers to preheat the liquor leaving the scrubbers. The process, which has been used in several coke-oven installations having a total production of about 3000 tons of ammonia per annum, has resulted in the yield of ammonia being increased from 0·14% to 0·21%, calculated on the weight of the dry coal, the cost of production of the ammonia being reduced by 20—30%. L. A. COLES.

Use of soaps and colloids for the purification of brine. J. WALTER (Bull. Soc. Ind. Mulhouse, 1926, **92**, 188—192).—Brine or pickling liquor is purified by treatment with an alkaline starch paste whereby colloidal inorganic impurities rapidly coagulate and settle so that the clarified brine can be removed by decantation. Similar methods of purification using black soap, potato and wheat starch pastes, and flour pastes are less satisfactory. The alkaline starch paste is prepared by heating together 6·0 g. of potato starch and 2·4 g. of caustic soda of *d* 1·384 at a temperature not exceeding 55° and this amount is added per cub. m. of brine.
A. J. HALL.

Colorimetric determination of nitrates. L. W. HAASE (Chem.-Ztg., 1926, **50**, 372).—In the colorimetric determination of nitrates by brucine and sulphuric acid in Autenrieth and Königsberger's wedge apparatus it is proposed to dissolve 5 g. of brucine in 100 c.c. of pure chloroform. The solution is slightly yellow, and may be kept without deterioration for weeks ; 0·2 c.c. is added to 10 c.c. of the liquid to be tested, and then 20 c.c. of concentrated sulphuric acid are added. The solution becomes heated, and most of the chloroform is evaporated. After cooling as quickly as possible the solution is made up to 30 c.c. with water free from nitrates and nitrites and compared with a standard in the wedge apparatus. The colour obtained is intense and persists for 24 hours. The reaction is quantitative with as little as 0·5 mg. of N_2O_5 per litre. Solutions containing more than 20 mg. N_2O_5 per litre must be diluted. A. RATNER.

Determination of chloride in commercial alkali cyanides. A. BOCK (Chem.-Ztg., 1926, **50**, 391).—The process depends on the fact that potassium cyanide is oxidised by hydrogen peroxide to cyanate and the latter decomposes yielding potassium hydrogen carbonate and ammonia, while any chloride present is not affected. A solution of 1 g. of the sample in 50 c.c. of water is warmed with 10—15 c.c. of 10% hydrogen peroxide free from chlorine, until ammonia is evolved. There should be no smell of hydrogen cyanide. The excess of hydrogen peroxide is then boiled off and the solution acidified with dilute nitric acid, when chloride can be determined as usual. Control tests gave good results. C. IRWIN.

Volumetric determination of hyposulphite. H. ROTH (Z. angew. Chem., 1926, **39**, 645—646).—A solution of hyposulphite is prepared by dissolving 3 g. of the salt in 5 c.c. of sodium hydroxide (*d* 1·4) and diluting to 250 c.c. A burette is filled with this solution and 2 c.c. of light petroleum coloured brown with asphalt are introduced to prevent oxidation. To 25 c.c. of water are added 1—2 c.c. of acetic acid and 1—2 g. of sodium bicarbonate to expel air, followed by 10 c.c. of sodium acetate solution (500 g. per litre), 0·1 g. of salicylic acid, and 10 c.c. of 0·1*N*-iron alum solution and the resulting red liquor is titrated with the hyposulphite solution until colourless. The procedure is repeated with the addition to the iron solution before titration of 0·15—0·17 g. of the hyposulphite to be analysed. The difference of the two readings of the burette is a measure of the iron reduced by the hyposulphite. Thiosulphate does not interfere in this method. A. R. POWELL.

Analysis of commercial bifluorides. E. C. ROPER and E. B. R. PRIDEAUX (J.S.C.I., 1926, **45**, 109—110 T).—The hydrofluoric acid contents of commercial potassium and sodium hydrogen fluorides determined by titration with alkali free from silicate, were 27·0% and 29·9% respectively. Sodium in the latter salt, determined as sulphate, was 37·32%, and total fluorine, determined as calcium fluoride, 57·75%. The salt contained 2·04% of water. On heating to a dull red heat the potassium salt evolved 88—89% of its hydrofluoric acid, and the sodium salt, at a slightly lower temperature, 81%.
A. B. MANNING.

Decomposition of mixtures and principle of physical substitution in the gaseous phase. [Preparation of hydrogen from water-gas.] G. CICALI (Giorn. Chim. Ind. Appl., 1926, **8**, 171—174).—A method is described for the economical preparation of the pure hydrogen necessary for synthesising both ammonia and liquid fuels, by the compression and partial liquefaction of water-gas and the like. Consideration of the pressure-temperature diagram of different hydrogen-carbon monoxide mixtures leads to the conclusion, which is confirmed experimentally, that, no matter what the liquefaction procedure adopted or the path traversed, the percentage of carbon monoxide present in the issuing hydrogen is invariably connected with the final state of the mixture. It is shown further that direct addition of carbon monoxide to water-gas or a similar gas would injure rather than improve the economic effect (the loss in hydrogen and the work of compression being increased) and the final effect of the purification. Also washing of the ascending gaseous phase by the liquid gradually condensing can never yield hydrogen devoid of carbon monoxide, even if, as Claude suggested, a suitable addition of nitrogen, instead of carbon monoxide, is made to the water-gas or similar gas prior to partial liquefaction of the mixture. On the basis of physical considerations relating to the properties of certain mixtures which are described as physically similar and from which a new principle known as physical substitution is deduced, the author suggests the introduction of a definite quantity

of gaseous nitrogen, not into the water-gas, but into a mixture derived therefrom, so as to give a mixture similar to, that is, having the same behaviour towards liquefaction as, water-gas. The few experimental results as yet available appear to confirm the theoretical results. T. H. Pope.

See also A., June, 581, **Catalysis of reaction between arsenious acid and permanganic acid and its analytical application** (Lang). 582, **Comparison of catalytic oxidation of hydrocyanic acid and ammonia** (Andrussow). 587, **Dissociation of dolomite** (Garnett). 588, **Influence of neutral salts on precipitation of metal sulphides** (Dede and Becker). 589, **Plumbic ammonium chloride** (Seyewetz and Tatu). 590, **Electrometric titration of halides** (Clark); **Possible error in calcspar determination of hydrochloric acid** (Rivett); **Volumetric determination of fluoride in simple, complex, and insoluble fluorides** (De Boer and Basart). 591, **Determination of arsenic** (Fridli); **Determination of carbon dioxide in carbonates** (Von Bruchhausen). 592, **Determination of mercuric chloride** (Von Bruchhausen and Hanzlik); **Air oxidation of titanous sulphate solution. Vanadous sulphate, a new and powerful reducing agent** (Russell); **Potentiometric standardisation of titanous chloride solution** (Zintl).

Patents.

Recovery of hydrochloric acid from ferrous chloride solutions. R. Ensoll (E.P. 251,495, 6.8.25).—Ferrous chloride solution containing free hydrochloric acid, *e.g.*, galvaniser's waste pickle, is gradually run into a heated retort of sulphuric acid kept at 115—125°, the process being continued until sufficient iron is present to form ferrous sulphate, which is discharged from the retort as a pasty mass. The evolved gaseous hydrochloric acid and water vapour are condensed and collected in the usual manner, the uncondensed gas being absorbed in a scrubbing tower. H. Royal-Dawson.

Production of hydrocyanic acid. R. W. Poindexter, Assr. to California Cyanide Co. (U.S.P. 1,584,137, 11.5.26. Appl., 2.12.19).—Hydrocyanic acid is produced by passing gaseous ammonia and a hydrocarbon through a zone heated at 1200° at such a rate that decomposition of ammonia and the reaction products is minimised. C. O. Harvey.

Production of additive compounds of hydrocyanic acid and metal chlorides. H. Stoltzenberg (E.P. 250,453, 15.8.25).—Additive compounds are prepared by the action of gaseous hydrogen cyanide, previously cooled or not, on an anhydrous metal chloride or a mixture of such chlorides, which is kept cool and stirred. The products are either dust-dry or granular and may be packed in tins immediately. T. H. Pope.

Production of sodium bicarbonate and sal ammoniac [ammonium chloride]. Ges. f. Kohlentechnik m.b.H. (E.P. 243,677, 21.9.25. Conv., 26.11.24. Addn. to 229,640; B., 1925, 880).—Gas liquors or other solutions of thiocyanates containing in addition varying amounts of thiosulphates, sulphates, polythionates, polysulphides, and organic impurities, and saturated with sodium and ammonium chlorides are treated with solid ammonium bicarbonate and stirred for about three hours at 30°, the precipitated sodium bicarbonate is filtered off, and the solution returned to the original bulk to be treated with fresh thiocyanate if necessary. Sodium chloride is added to the filtrate in quantity equivalent to the ammonium bicarbonate, and stirred for some time, lowering the temperature about 10°, thus causing the separation of ammonium chloride. H. Royal-Dawson.

Producing alkali sulphides. G. de Béthune and R. Vahrenkampf, Assrs. to Soc. Nationale d'Ind. Chim. en Belgique, S. A. (U.S.P. 1,578,718, 30.3.26. Appl., 16.1.24).—A saturated solution of sodium nitrate at 25° is stirred with an equivalent amount of barium sulphide, either powdered or as a saturated solution, in a closed vessel containing hydrogen sulphide under a pressure of about 1 atm. The temperature is maintained at 110° for $\frac{1}{4}$—1 hr. and the mass is then cooled slowly. The barium nitrate solution is separated by decantation from sodium hydrogen sulphide in the presence of hydrogen sulphide. Addition of a little carbon disulphide facilitates this separation. The sodium hydrogen sulphide is then heated to convert it into sodium sulphide. R. B. Clarke.

Process of making sodium sulphide. H. P. Bassett (U.S.P. 1,584,597, 11.5.26. Appl., 26.8.25).—Sodium sulphate is heated with carbonaceous matter at about 650° until 20—25% of the theoretical yield of sodium sulphide is obtained; the temperature is then raised to about 700°, yielding 40—60% of the theoretical amount, and finally further raised to about 900°. H. Royal-Dawson.

Conversion of sodium sulphide and similar substances into a granular form. B. Roos & Co. (G.P. 426,052, 19.1.24).—The melted material is caused to fall from a suitable height (either in an intermittent or continuous stream) into a liquid, such as trichloroethylene, which has a lower b.p., and does not react with the substances used. A. Coulthard.

Recovery of thiocyanates. M. Darrin, Assr. to Koppers Co. (U.S.P. 1,584,852, 18.5.26. Appl., 16.1.23).—Thiocyanate is recovered and separated from an impure solution in the form of sodium thiocyanate hydrate, $NaCNS,2H_2O$. H. Royal-Dawson.

Production of sodium and potassium cyanides. E. Hene (G.P. 427,156, 11.1.24).—Solutions containing calcium cyanide or mixtures forming it are treated with alkali sulphates in such quantity that calcium sulphate free from syngenite settles out, and is removed. The solution is then treated with excess of alkali sulphate or carbonate to complete the conversion of the calcium cyanide into alkali cyanide,

and the alkali sulphate in the precipitated syngenite is recovered by extraction with water, or is utilised for the decomposition of more calcium cyanide.
L. A. COLES.

Production of ferric oxide. O. S. NEILL (E.P. 251,310, 24.10.24).—Solutions of an iron compound, *e.g.*, waste pickle liquors, are sprayed or atomised through a fireproof chamber into which is led a current of heated air or gas, or a mixture of both, in the same downward direction. On withdrawing the moist air or gas by means of a fan, the dehydrated product is introduced while still hot into a rotary furnace and heated to about 200°; it is finally calcined at 500—900° in air or oxygen for conversion into iron oxide of 90% purity, and the gaseous by-products are recovered.
H. ROYAL-DAWSON.

[Apparatus for] making oxidised leaden powder [litharge]. C. A. HALL (E.P. 251,449, 26.5.25).—The apparatus is designed for carrying on the process in which leaden balls are oxidised by the action of a current of air in a rotating vessel. The latter consists of a cylindrical central portion with ends of frusto-conical form, and is on an axis slightly inclined to the horizontal in the direction of the feed. It is provided with exterior water cooling, an enclosed feed for the lead, and a delivery hopper with a screen for separating the fines from any coarser product.
C. IRWIN.

Slaked lime. C. E. HITE (U.S.P. 1,579,766, 6.4.26. Appl., 7.8.25).—Quicklime is slaked with a lime–sulphur solution prepared by heating together 1 lb. of quicklime, 2 lb. of sulphur, and 1 gal. of water to produce a solution of *d* 1·25 approximately.
E. S. KREIS.

Manufacturing hydrated lime. C. I. CHUBBUCK (U.S.P. 1,580,710, 13.4.26. Appl., 28.2.24).—Quicklime is slaked with excess of water so that the temperature does not exceed 100°, and the sludge formed, which contains 50—90% of water, is rapidly filtered to give a paste containing 25—40% of water. The filtrate is employed to slake fresh lime and the lime paste is conveyed to a bin where hydration is completed. The heat produced evaporates the remainder of the water and a dry hydrated lime is obtained.
T. S. WHEELER.

Process of treating lime. F. C. MATHERS, Assr. to NATIONAL LIME ASSOC. (U.S.P. 1,583,759, 4.5.26. Appl., 22.8.22).—Quicklime is slaked to a dry hydrated lime by treating it with an equal weight of a 1% solution of calcium chloride or other alkaline-earth chloride. The product on treatment with excess of water yields a suspension which settles more rapidly than material similarly prepared by slaking with water.
T. S. WHEELER.

Manufacture of carbon monoxide. F. JOURDAN and J. GALL (F.P. 601,998, 13.11.24).—Excess carbon is burnt in air enriched with oxygen below 1000°.
A. COULTHARD.

Manufacture of phosphoric anhydride. I.-G. FARBENIND. A.-G., Assees. of G. PISTOR (G.P. 426,388, 31.12.24).—Phosphorus or a gaseous mixture containing phosphorus is burnt by means of air which has been previously dried with phosphorus pentoxide or phosphoric acid. The drying agents are produced in the process itself. The heat arising from the combustion is utilised for heating a steam boiler or other apparatus.
A. COULTHARD.

Manufacture of hydrogen. W. P. ROGERS (E.P. 251,124, 29.5.25).—In the manufacture of hydrogen from iron and steam, the material is placed in a cast-iron retort which is rectangular in cross-section, having one side longer than the other; the gases are caused to flow through the retort in a direction transverse to its longer axis.
H. ROYAL-DAWSON.

Separation of hydrogen from water-gas, coke-oven gas and similar gases. H. M. R. BARJOT (F.P. 601,774, 7.5.25).—The gas under treatment is allowed to circulate along a porous wall, on the other side of which there is an evacuated vessel in which nitrogen may circulate and take up the hydrogen diffusing through the wall.
A. COULTHARD.

Manufacture of hydrogen. F. JOURDAN and J. GALL (F.P. 601,997, 13.11.24).—Carbon monoxide is catalysed under pressure in the presence of a large quantity of steam.
A. COULTHARD.

Method and apparatus for liquefying chlorine. W. M. JEWELL, Assr. to CHLORINE PRODUCTS CO. (U.S.P. 1,584,376, 11.5.26. Appl., 11.2.21).—The gas is compressed and condensed and, without admixture with air, led into evacuated containers at a lower temperature than that of the liquefied gas during condensation.
H. ROYAL-DAWSON.

Production of phosphoric acid anhydride. CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 245,430, 8.12.25. Conv., 30.12.24).—See G.P. 426,388; preceding.

Recovering gaseous constituents [sulphur dioxide] from gas-mixtures. M. SCHROEDER (E.P. 251,805, 10.7.25).—See G.P. 421,725; B., 1926, 321.

Sulphite liquor (G.P. 426,386).—See V.

Nitric oxide by the electrical method (F.P. 601,740).—See XI.

VIII.—GLASS; CERAMICS.

Influence of boric oxide on the properties of chemical and heat-resisting glasses. II. Resistance to chemical reagents. W. E. S. TURNER and F. WINKS (J. Soc. Glass Tech., 1926, **10**, 102—113; cf. B., 1926, 238).—A series of glasses obtained by replacing silica, stage by stage, by boric oxide in a Kavalier (soda–potash–lime–silica) glass, was tested for durability. Towards boiling water the resistance remained constant until a boric oxide content of 10·78% was reached, after which durability rapidly decreased. Boiling hydro-

chloric acid gave constant attack with a boric oxide content up to 2%, slowly increasing attack from 2 to 7·9%, and rapidly increasing attack above this point. With boiling sodium hydroxide the attack was constant up to 2% of the oxide, from thence slowly increasing up to 13·65%, beyond which a rapidly increasing attack occurred. Boiling water extracted silica and lime in a proportion slightly less, and boric oxide and alkalis slightly greater, than from the original glass. Boiling hydrochloric acid extracted from glasses rich in boric oxide all the constituents but the silica, which was left entirely as a skeleton of the shape of the glass.

A. COUSEN.

Effect of composition on the viscosity of glass. IV. Calculation of the influence of minor constituents. S. ENGLISH (J. Soc. Glass Tech., 1926, **10**, 52—62; cf. B., 1925, 715).—By a series of successive approximations the author eliminated the effect of minor constituents on the viscosity of glasses previously examined. From factors for major constituents so obtained he then compared the viscosity of a soda–lime glass with that of a similar glass, as determined by Stott (B., 1925, 449), agreement proving to be close, especially over the range 1300—1100°.

A. COUSEN.

Function of arsenic in soda-lime-silica glasses. I. E. M. FIRTH, F. W. HODKIN, M. PARKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1926, **10**, 3—19).—Arsenious oxide, added to a soda–lime batch to the extent of 10 parts per 1000 of sand, remains practically completely in the glass when melting takes place at 1400° in closed pots. With 250 parts of the oxide per 1000 of sand at least 60% is retained. Even in presence of no oxidising agent 40 to 70% of the arsenic is converted during melting into arsenic pentoxide. The arsenious oxide exerts no beneficial effect on melting, and fining is not assisted by amounts up to 10 parts per 1000 of sand, whilst large amounts of the oxide raise the melting temperature. In batches rich in silica, addition of more than two parts of arsenious oxide per 1000 of sand tends to cause a surface scum. The green tint due to iron in the glass is reduced by the arsenious oxide, improvement being continuous with increasing proportions of arsenic. When the oxide is added to the extent of 150 to 250 parts per 1000 of sand an opalescent glass is produced.

A. COUSEN.

Devitrification of soda-lime-silica glasses containing excessive amounts of arsenious oxide. M. PARKIN and W. E. S. TURNER (J. Soc. Glass Tech., 1926, **10**, 20—26).—The devitrification temperatures of glasses made from soda–lime batches containing 0, 20, 25, 50, 100, 200, and 250 parts of arsenious oxide per 1000 of sand respectively showed a minimum (575° to 600°) when 100 parts of the oxide were employed. Devitrification of the higher members was probably hindered by the increased hardness of the glass. No definite determination of devitrification temperature could be obtained by the method of heat evolution. Microscopical examination indicated spherulitic devitrification crystals of uncertain character. A. COUSEN.

Composition of glass suitable with automatic glass-forming machines. W. E. S. TURNER (J. Soc. Glass Tech., 1926, **10**, 80—91).—A number of glass compositions and batch formulæ for various uses such as with sheet-glass machines, automatic tube-drawing machines, and automatic machines for containers, are quoted. Practical and scientific factors governing the choice of batch for these purposes are also considered. A. COUSEN.

Expansion of industrial glasses. M. SAMSOEN (Compt. rend., 1926, **182**, 1384—1386).—Three typical glasses rich in lime, alkali, and alumina respectively, were mixed in varying proportions, fused at 1400—1500°, and cast in rectangular rods. The coefficient of expansion at 20°, the transition temperature, and the coefficient of expansion above the transition temperature were determined. The simple additive law of Winkelmann and Schott does not apply to these mixtures. The coefficient of expansion increases with rise of temperature, and is very much greater above the transition temperature than below it. The compositions of the glasses varied within the following limits: Al_2O_3, 1·7 to 6·37%; CaO, 8·6 to 17·35%; (Na_2O+K_2O), 10·0 to 18·3%; SiO_2, 66·1 to 69·95%. W. HUME-ROTHERY.

Method of recognising chemically inferior glasses. F. FRIEDRICHS (Z. angew. Chem., 1926, **39**, 611).—The surface of inferior glass swells when it is boiled with water or steamed. If, after treatment for 12 hrs., the glass is rapidly heated until it begins to soften, the swollen surface dries and cracks. After heating for 2 hrs. the surface of very poor glass has a matt appearance; in the case of somewhat better glass the surface cracks are readily visible at a magnification of 100—200 diameters, whilst good glass remains unaffected by the treatment.

A. GEAKE.

Development of sillimanite refractories for glass making. W. A. MCINTYRE (J. Soc. Glass Tech., 1926, **10**, 73—80).—A process of weathering of clay–sillimanite mixtures for six weeks improved the workability of the mixture and the bonding of the two constituents. As a bonding agent, Stourbridge O.M. clay gave the strongest bonding with least soaking at all firing temperatures. Fine-grained mixtures most successfully withstood corrosion, and in a series of small pot meltings the only sign of glass attack was along the flux line. Details of the successful use of a sillimanite–fireclay (70 : 30) mixture as a pot-furnace siege are given.

A. COUSEN.

See also A., June, 575, **Colloid chemistry of clay and kaolin** (NISHIKAWA). 576, **Anomalous flocculation of clay** (JOSEPH and OAKLEY).

PATENTS.

Annealing glass. K. M. HENRY, Assr. to ILLINOIS-PACIFIC GLASS CO. (U.S.P. 1,585,542, 18.5.26. Appl., 25.11.24).—Glassware is subjected

to a series of stages of successively falling temperature within the annealing range, in a temperature-controlled lehr or muffle. A. Cousen.

[Magnesite] refractories. U.S. Metals Refining Co., Assees. of A. Marks (E.P. 244,391, 21.1.25. Conv., 10.12.24).—Dried magnesite is mixed with a drying or semi-drying animal or vegetable oil, *e.g.*, raw linseed oil, with the addition of a fluxing material if necessary, and fired in the usual manner. The refractory material produced is denser and less porous than normal magnesite products, with a lower coefficient of heat expansion and a longer life under working conditions. B. W. Clarke.

Refractory composition. W. A. Farish (U.S.P. 1,561,641, 17.11.25. Appl., 31.5.18).—A cheap refractory material of low coefficient of expansion and high chemical resistance is prepared by pressing a mixture of 65 parts of silicon carbide, 32 parts of tar (or other carbonising binder), and 3 parts of borax or sodium silicate, and baking it at 1000°. R. B. Clarke.

Process for enamelling metal articles. R. A. Auger (E.P. 251,327, 27.1.25).—The process, particularly suitable for brass and gun-metal water taps and cocks, consists in coating the non-ferrous metal or alloy with a thin homogeneous skin of copper or other suitable metal, preferably by electrolytic deposition, and then heating to redness (about 900°) and slowly cooling, after which the article is enamelled and finally annealed. A. Cousen.

Manufacture of synthetic emerald stones. J. F. Riera (U.S.P. 1,579,033, 30.3.26. Appl., 4.10.24).—Rock crystal (90 g.) is heated to redness, rapidly cooled in water, and pulverised; the product is treated with an aqueous solution of copper and uranium nitrates, mixed with 20 g. of alumina, 20 g. of anhydrous or hydrated beryllia, and a flux consisting of 30 g. of lithium carbonate, 15 g. of fused boric acid, and 4 g. of fused sodium borate, and the mixture melted in an electric furnace at 1600—1900° and afterwards broken up into fragments of the required size. These are softened by heating in a muffle at 600—800° and moulded or compressed to imitate the appearance of natural emeralds. R. B. Clarke.

Salt glazing. H. G. Schurecht (U.S.P. 1,583,901–2, 11.5.26. Appl., 23.7.24).—(a) An alkali chloride together with a volatile material containing zinc or (b) potassium chloride is volatilised in the kiln during the burning of the ware. B. W. Clarke.

Scum prevention [on clay ware]. H. G. Schurecht (U.S.P. 1,583,903, 11.5.26. Appl., 23.7.24).—A precipitant is added to the clay which will react with the soluble salt liable to form scum on the ware, to form insoluble and soluble salts, the latter being rendered insoluble by a second precipitant. B. W. Clarke.

Tunnel kiln. P. A. Meehan and H. M. Robertson, Assrs. to Amer. Dressler Tunnel Kilns Co. (U.S.P. 1,584,883, 18.5.26. Appl., 30.6.22).—In a continuous tunnel kiln the ware is transported by a train of cars, each consisting of a metallic underframe on wheels and a refractory superstructure, having a longitudinal cavity in its upper face, in line with the cavities of the other cars, and so forming a continuous trough, extending the length of the train. A. Cousen.

Tunnel kiln. G. W. Booth (U.S.P. 1,585,013, 18.5.26. Appl., 19.2.25).—A tunnel kiln of the ordinary type comprising preheating, firing, and cooling chambers has two drying chambers which extend the length of the kiln above the other three chambers and are heated by conduction from these chambers, and by the products of combustion from the kiln furnace which pass through flues alongside the drying chambers. Air is drawn in through flues adjacent to the cooling chamber in which it cools the ware and is preheated and is then passed into flues which extend alongside the drying chambers and communicate with them at intervals. The hot air passes from these flues into the drying chambers, and the moist air is led thence into an exhaust flue and discharged. The material to be treated passes through one or other of the drying chambers and is lowered at the discharge end to the level of the preheating chamber into which it is introduced. T. S. Wheeler.

[Tunnel] kiln for burning earthenware. W. L. Burley (U.S.P. 1,585,015, 18.5.26. Appl., 15.1.25).—The kiln consists of preheating, baking, and cooling zones, and has fire-boxes on opposite sides of the tunnel adjacent to, and discharging into, the baking zone. In the side walls of the kiln and fire-boxes are longitudinal flues which extend over the greater part of the preheating and cooling zones, communicating near the front of the preheating zone with an adjacent stack. The flues are open to the air at the cooling ends, but closed at the ends in the preheating zone and they are not in direct communication with the fire-boxes. Transverse flues in the side walls of the preheating and cooling zones lead from the tunnel to the longitudinal flues. A. Cousen.

Casting slip. H. T. Bellamy, Assr. to Western Electric Co. (U.S.P. 1,585,010, 18.5.26. Appl., 8.10.23).—50 pts. of silicon carbide ground to 220-mesh are mixed with 12 pts. of Florida clay, 12·5 pts. of English china clay, 1/8th part of solid sodium carbonate, 3/8th part of liquid sodium silicate, and 40 pts. of water to give a mixture of *d* 2·25, and the slip obtained is cast and fired. The product is a refractory which will stand rapid variations in temperature, is a good conductor of heat, and a good electrical insulator even at high temperatures. It is especially adapted to the manufacture of supports for the heating wires of electric soldering irons. T. S. Wheeler.

IX.—BUILDING MATERIALS.

Ferruginous and aluminous cements. E. Martin (Mon. Sci., 1926, [v], **16**, 97—101).—Ferruginous cements, consisting chiefly of calcium

ferrites, $2Fe_2O_3,nCaO$, (n=5, 6, or 7), can be obtained by heating the raw materials (lime and, *e.g.*, pyrites cinder) together below the fusion temperature. These cements set slowly but are very resistant to attack by waters containing sulphates and alkalis. Ferro-aluminous cements consisting of mixtures of calcium ferrites, aluminates, and ferro-aluminates, are made in a similar way; by varying the proportions of ferric oxide or alumina, the rate of setting and also of hardening can be varied within wide limits. All the cements are very resistant to the attack of sulphates, and are suitable for reinforced concrete work in sea water. B. W. Clarke.

Cement for floors and plastering on walls. F. Greenbaum (Chem.-Ztg., 1926, 50, 409—410).—Filling materials, *e.g.*, sand, clay, sawdust, kieselguhr, etc., are mixed in varying proportions with a magnesium oxychloride cement consisting of about 6 pts. by wt. of magnesium oxide to 1 pt. of magnesium chloride, forming products of which the strength, water-resisting properties, etc. depend on the filling materials used. The following mixture is recommended: magnesium oxide 60, sand 30, kieselguhr 12, clay 12, magnesium chloride 9, water 45 pts. by wt. B. W. Clarke.

Constitution of magnesium oxychloride cement. T. Maeda and S. Yamane (Sci. Papers Inst. Phys. Chem. Res., 1926, 4, 85—101).—In order to elucidate the constitution of magnesian cements the system $MgO-MgCl_2-H_2O$ was studied at 50° in a special apparatus. The solid phases MgO, H_2O and $3MgO,MgCl_2,12H_2O$ can coexist in stable equilibrium with solutions containing 14·01% of magnesium chloride, whilst at 37% the double salt and $MgCl_2,6H_2O$, form the solid phase. MgO,H_2O can remain in unstable equilibrium with solutions containing from 14·01 to 20·95% of magnesium chloride, above which only the double salt exists. Below 12·45% the double salt decomposes, but it may remain in unstable equilibrium between 12·45 and 14·01% of magnesium chloride. On the basis of the above results a formula for the calculation of the composition of various samples of cement is derived. It appears that ordinary magnesian cement consists of the double salt, magnesium oxide monohydrate, and solution. The proportion of the first constituent is fairly constant (57·8—61·8%), that of the others varying more widely. The content of solution may attain 34·1%. R. Truszkowski.

Viscosity changes in the reaction between magnesia and aqueous magnesium chloride, and the setting of cement. T. Maeda (Sci. Papers Inst. Phys. Chem. Res., 1926, 4, 102—128).—The rate of increase of viscosity of mixtures of magnesium oxide with aqueous solutions of magnesium chloride was studied under various conditions, using a modification of Ostwald's apparatus. The viscosity increases rapidly with rise of temperature and with increase in concentration of magnesia. The higher the temperature at which the magnesia used was calcined, the longer is the time required for setting. Greater concentration of magnesium chloride also delays setting, whilst the addition of sodium chloride or gelatin has little influence. In certain cases irregularities in the shape of the curve of increase in viscosity with time were observed, and these are shown to be due to supersaturation of magnesia in the solution and to crystallisation of oxychloride, followed by colloidal hydration. From the results obtained it is concluded that in the setting of cements and plasters the liquid phase is supersaturated with respect to the solid, which crystallises out in small aggregates, on which water is then adsorbed. R. Truszkowski.

Patents.

Treatment of concrete surfaces. N. C. Johnson (E.P. 228,538, 26.1.25. Conv., 2.2.24).—A reagent, *e.g.*, sugar or tannic acid, which will prevent the setting of cement with which it comes into contact, is dispersed in a viscous colloidal solution, *e.g.*, starch, and applied to the forms or to freshly laid concrete to improve the appearance of the concrete surface. B. W. Clarke.

Process for making cement from town and like refuse. G. E. Heyl (E.P. 251,558, 14.12.25).—Refuse dried by waste heat is ground and mixed with ingredients such as lime and bauxite which form a cement-making mixture with the ash of the refuse, and the mixture is burnt in a cement kiln, the waste heat of which may be utilised for drying the refuse. B. W. Clarke.

Manufacture of an artificial gypsum stone. M. Claasz (G.P. 426,760, 3.4.25).—Dolomite is treated with sulphur dioxide and the product is mixed with gypsum. A. R. Powell.

Bituminous emulsions [for treating roads]. W. J. McGivern, J. H. Foster & Co., Ltd., and R. Swift (E.P. 251,098, 29.4.25).—A protein or similar colloidal substance, *e.g.*, albumin, casein, etc., is mixed thoroughly with molten bitumen and a soap of a vegetable oil or fatty acid or similar products kept at a temperature of 125—130°. B. W. Clarke.

Process for colouring wood. Badische Anilin- & Soda-Fabr., Assees. of K. Immerheiser and K. Neubauer (G.P. 422,124, 27.6.24).—Solutions of dyes are used which contain both acid and basic colours dissolved in a mixture of water and an organic solvent miscible with water, such as acetone, ethylene chlorohydrin, acetin, or mixtures of these. This avoids the mutual precipitation which frequently occurs when a mixture of both acid and basic dyes is dissolved in water alone. The wood is dyed in different shades lying side by side, producing a multicolour effect. For example, an aqueous solution of Naphthol Yellow S and a solution of Methyl Violet in ethylene chlorohydrin dye wood, under pressure, partly red and partly black; wood treated with aqueous solutions of Green PLX and Naphthol Yellow S and Fuchsin powder in acetone shows yellow, red, and black shades; solutions of Ponceau in water and Diamond Green in acetin give a red and black spotted effect. Wood under pressure, is dyed partly yellow and partly blue when treated with a green mixed solution of Water Blue IN in water and Auramin II in ethylene chlorohydrin. A. Coulthard.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of the casting temperature and of annealing on the magnitude and distribution of the phosphide eutectic [in cast iron]. H. Jungbluth and H. Gummert (Kruppsche Monatsh., 1926, **7**, 41—46; Chem. Zentr., 1926, I., 3275).—The amount of the phosphide eutectic in cast iron is smaller the lower the casting temperature. Annealing above 700° also decreases the amount of eutectic, as above 700° phosphorus rapidly diffuses into the ferrite constituent. The casting temperature has no effect on the grain structure but annealing coarsens it.
A. R. Powell.

Determination of sulphur in iron and steel. A. Kling and A. Lassieur (Chim. et Ind., 1926, **15**, 699—701).—The evolution method of determining sulphur in cast iron and steel gives results as accurate as those obtained by gravimetric methods provided that the metal is dissolved sufficiently rapidly to prevent absorption of organic compounds from the gas stream by the zinc acetate solution. The acid used should be made by mixing 3 vols. of hydrochloric acid (*d* 1·19) with 1 vol. of water, and the carbon dioxide stream should be interrupted during the actual dissolution of the metal, being used solely to drive the air from the apparatus before starting and to remove the gas retained in the dissolving flask after finishing the analysis.
A. R. Powell.

Electrolytic determination of nickel in nickel steel. W. Moldenhauer (Z. angew. Chem., 1926, **39**, 640—642).—Electrolysis of ammoniacal nickel solutions containing ferric hydroxide in suspension invariably leads to contamination of the nickel deposit with more or less iron, but the deposition of nickel is quantitative in 1 hr. if a rotating gauze cathode is used. To obtain correct results for nickel the deposit should be dissolved in dilute sulphuric acid and hydrogen peroxide and the electrolysis repeated after addition of ammonia. In the presence of chromium hydrazine sulphate should be added to avoid oxidation of the deposit by the chromic acid formed at the anode. In both electrolytic operations 15 g. of ammonium sulphate must be present in the solution, which is preferably maintained at 60—70°. The method is suitable for the rapid determination of nickel in steel provided that cobalt is absent; if present it is deposited quantitatively with the nickel and must be subsequently separated by any of the usual methods.
A. R Powell.

Resistivity and thermo-electric power of reversible iron-nickel alloys from —200° to +1000°. P. Chevenard (Compt. rend., 1926, **182**, 1388—1391).—The resistivity of iron-nickel alloys has been determined from —200° to +1000°, and also the thermo-electric power against a wire of a platinum alloy. The magnetic transformation of the reversible alloys is shown by a change in direction of the particular isothermal. The line joining the points on the different isothermals shows a slight change in direction at the composition Fe_2Ni, but the existence of this as a definite compound is not conclusively established.
W. Hume-Rothery.

Formation of powdered copper in anode mud. M. de K. Thompson (Chem. Met. Eng., 1926, **33**, 298).—The presence of finely-divided copper in the anode mud from copper refining is due to the reaction $2\ Cu^{\cdot} \rightleftharpoons Cu + Cu^{\cdot\cdot}$ proceeding from left to right. It is shown theoretically that this behaviour cannot be caused by dilution of the anolyte as that should increase the cuprous ions, but is caused by the diffusion of the warm layers of liquid next to the anode (and therefore saturated with cuprous ions) into the cooler main body of solution; the resulting drop in temperature causes metallic copper to be precipitated. A similar explanation accounts for the presence of gold in anode mud from gold refining. A. R. Powell.

Cementation of copper, nickel, and their alloys by tin. L. Guillet (Compt. rend., 1926, **182**, 1363—1365).—The use of molten tin for the cementation of copper alloys causes a surface deterioration, which may be prevented by using a copper-tin alloy (25% Sn) at 700°, with ammonium chloride as a flux. The cementation has been examined with copper, nickel, brasses, bronze, cupro-nickel, and nickel-silver. Very little cementation occurs with pure copper or nickel but with the other alloys the surface layer varies from 0·35 to 1·0 mm. after treatment for 48 hrs. at 700°. The cemented layer usually contains the typical constituents of copper alloys which can be identified microscopically. In the case of cupro-nickel (80% Cu) the surface layers contain a solid solution showing numerous slip-bands. The method enables a malleable alloy after shaping to be given a surface with the properties of an alloy which is hard and resistant but not itself malleable.
W. Hume-Rothery.

Hardening of lead-antimony, lead-tin, and lead-antimony-tin alloys. L. Guillet (Compt. rend., 1926, **182**, 1362—1363).—The hardening of lead-antimony alloys by quenching described by Dean, Zicheick, and Nix (Amer. Inst. Min. Eng., Feb., 1926) had previously been noted by Dubosc (Réunion de l'Assoc. Int. pour l'essai des matériaux de construction, 1905), whilst the present author had already described experiments on lead-tin alloys (Chim. et Ind., 1921, **5**, 371; cf. B., 1922, 297 A).
W. Hume-Rothery.

Determination of tin in non-ferrous alloys. H. N. Marr (Metal Ind., 1925, **27**, 77—78).—The alloy is oxidised with nitric acid (*d* 1·2), the solution evaporated, and the dry residue treated with dilute nitric acid. The residue of tin and antimony oxides is ignited with zinc filings and zinc oxide, the mass dissolved in hydrochloric acid, the solution reduced with nickel, and titrated with iodine solution.
A. A. Eldridge.

Constitution of ternary systems [alloys]. W. Guertler (Z. anorg. Chem 1926, **154**, 439—455).—A detailed account of the means of differentiation between the most valuable metal

combinations and those which are of little technical use. Three points of view are specially considered: (i) The molten mixture must be a homogeneous, completely miscible mass. (ii) The mixture must have all the technically important properties of metallic substances—ductility etc. (iii) There must be a certain stability towards air and moisture. A scheme is very fully expounded for the graphical representation of the composition of ternary mixtures and an explanation of the use of these graphs for deducing the constitution of mixtures, *i.e.*, formation of mixed crystals, solid solutions, etc. The above is illustrated by special reference to previously described aluminium–magnesium–zinc, antimony–nickel–sulphur, and copper–antimony–tin systems. M. Carlton.

Theory of and investigations into the corrosion of metals. W. Palmaer (Korrosion u. Metallschutz, 1926, 2, 3—8, 33—38, 57—62; Chem. Zentr., 1926, I., 3360).—The velocity of dissolution of metals in acids and in salt solutions is governed by the difference in potential, by the conductivity of the solution, and by the resistance capacity of the local cells. To explain the induction period during which the rate of dissolution increases to a maximum and then falls again, it is pointed out that, at the beginning of the action, there are only a small number of points where the more noble impurity is exposed but, after some time, the amount of the less noble metal dissolved results in a greater exposure of surface of the impurity. To account for the fact that an absolutely pure and homogeneous metal is not attacked by corroding media the theory is advanced that a metastable condition is set up in which the whole of the surface has the same potential against the solution and therefore no electrolytic action can take place. Experiments are described which show that carbon dioxide alone has no appreciable action on the rate of rusting of iron; atmospheric oxygen, however, acts as a depolariser and assists the action of the carbon dioxide. The electrical conductivity of the liquid film on the iron appears to be the controlling factor in causing rusting to take place.
A. R. Powell.

Chemical equilibrium between lead sulphide and its oxidation products. R. Schenck (Z. anorg. Chem., 1926, 153, 149—152).—A reply to Jaenecke's criticism (cf. A., 1926, 358) of earlier papers (B., 1925, 287, 995). A. Geake.

Analysis of copper – palladium – gold – silver concentrates. C. W. Davis (U.S. Bur. Mines Rep. Investigations, 1926, [2731]; Chem. Abstr., 1926, **20**, 1365).—The material is fused with lead oxide, borax, and sodium carbonate. For the palladium determination, silver is added. After addition of lead, the buttons are scorified and cupelled, the beads parted with nitric acid, and the gold is weighed. The silver from a separate quantity is precipitated as chloride, the filtrate evaporated to dryness with hydrochloric acid, and the residue moistened with hydrochloric acid and filtered. The palladium is precipitated with dimethylglyoxime, and ignited in hydrogen. A. A. Eldridge.

Determination of chromium in chromite. A. Franke and R. Dworzak (Z. angew. Chem., 1926, **39**, 642—644).—One gram of the finely-ground mineral is heated with 8 g. of sodium peroxide in a covered porcelain crucible for 15 min., avoiding fusion. The mass is then just fused, allowed to cool, and extracted with boiling water. After boiling for 10 min. the solution is acidified with sulphuric acid and filtered into a 500-c.c. graduated flask; 100 c.c. of the filtrate are treated with 25 c.c. of 5% ferrous sulphate solution and 5 c.c. of sulphuric acid and the excess ferrous salt is titrated with permanganate to the first visible colour change. The permanganate is preferably standardised against potassium dichromate as the iron or oxalic acid figures give slightly low results. Good results are also obtained by the iodometric method provided that the alkaline chromate solution is evaporated to dryness, after filtering off the bulk of the iron, so as to render the last traces of iron insoluble. The presence of this iron in the filtrate is the cause of the slightly high results obtained usually by this method. A. R. Powell.

See also A., June, 562, **Crystal structure of duralumin** (Anderson) 563, **Single-crystal tungsten wire and theory of recrystallisation** (Ohashi). 564, **Recrystallisation and improvement of crystalline structure of metals** (Masing); **Deformation lines in large and small crystals of ferrite** (O'Neill); **Relation between lattice constant and density of iron-nickel alloys** (Osawa). 565, **Constitution of alloys of aluminium and magnesium with 32—48% Mg** (Halstead and Smith). 566, **Magnetic changes in iron and steel below 400°** (Dearden and Benedicks); **Specific resistance and thermo-electric potential of steels differing only in carbon content** (Campbell and Mohr). 581, **Liquid-line corrosion** (Hedges). 589, **Preparation of pure alkali metals** (Suhrmann and Clusius); **Germanium and gallium from germanite** (Kiel). 591, **Microchemical determination of lead** (Geilmann and Höltje). 592, **Iodometric determination of lead, cadmium, and mercury** (Kraus); **Gravimetric determination of copper as cuprous iodide** (Kolthoff and Kuylman); **Electrolytic determination of copper in presence of bismuth** (Moldenhauer); **Separation of metals of group III.** (Longinescu and Chaborski). 593, **Phenylarsinic acid as reagent for determination of zirconium and thorium** (Rice, Fogg, and James).

Patents.

Steel manufacture. T. J. Bray (U.S.P. 1,572,179, 9.2.26. Appl., 5.6.22).—The steel is deoxidised in the furnace and a small quantity ($\frac{1}{2}$ lb. per ton of steel) of sodium carbonate added as the steel is run into the moulds. The artificial effervescence thus produced throughout the body of the metal in the mould is stated to produce a high grade of steel.

The sodium oxide produced acts as a flux, and assists the removal of impurities. E. S. KREIS.

Separating the components of alloys. C. G. BOSSIÈRE and H. ZANICOLI (E.P. 241,880, 2.10.25. Conv., 23.10.24).—An alloy containing copper, lead, tin, and antimony is treated for recovery of its component metals with a mixture of sulphur and an alkali sulphide, polysulphide, or thiosulphate. The mixture so obtained is lixiviated with water, whereby soluble sulpho-salts are separated from insoluble sulphides, which are then roasted. The sulphur dioxide evolved is used to precipitate sulphides and sulphur from the solutions of the sulpho-salts. This mixed precipitate is then heated in a closed vessel and the sublimed sulphur is used in the initial treatment of the alloy. The sulphides remaining after sublimation of the sulphur are roasted to oxides, and the sulphur dioxide so formed is utilised to precipitate further quantities of sulpho-salt solution. The solution remaining after treatment of the sulpho-salts is evaporated, and the residual alkali salts are reduced to sulphide by roasting with carbon. L. M. CLARK.

[Non-corrosive] alloys. R. WELLESLEY (E.P. 250,721, 26.2.25).—A non-corrosive alloy, which can be cast, rolled, or stamped, is obtained by adding 1 pt. of an alloy of tin (16 pts.) and bismuth (1 pt.) to a molten mixture of copper, 40·5 pts., nickel 31·5 pts., zinc 25·5 pts., and aluminium 1 pt. The alloy is covered with charcoal during manufacture to prevent oxidation. L. M. CLARK.

White gold [alloy]. D. BELAIS, Assr. to D. BELAIS, INC. (U.S.P. 1,584,352, 11.5.26. Appl., 30.4.21. Renewed 30.9.25).—A white gold alloy contains more than 50% Au with smaller amounts of nickel and zinc and a very small proportion of manganese. A. R. POWELL.

Magnesium–aluminium alloy. H. E. BAKKEN, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,584,688, 11.5.26. Appl., 25.8.20).—The alloy is composed mostly of magnesium with a smaller quantity of aluminium, and manganese about 1%, but in any case not more than the aluminium. The sp. gr. is near that of magnesium. B. M. VENABLES.

High-lead bronze alloy and method of manufacture. W. E. DAY, JUN., Assr. to INTERNATIONAL MOTOR Co. (U.S.P. 1,584,706, 11.5.26. Appl., 20.10.23).—A mixture of lead and copper is heated to at least 1480°, the requisite amount of tin and a deoxidiser are then added, and the alloy is poured into ladles for casting into moulds. A. R. POWELL.

[Lead–antimony] alloy. J. GEPPERT (U.S.P. 1,584,922, 18.5.26. Appl., 6.10.22).—An alloy comprising 62—72 pts. of lead, 16—24 pts. of antimony, 2—16 pts. of bismuth, and 0·5—7·5 pts. of cadmium is claimed. A. R. POWELL.

Bearing metal with bronze base. T. GOLDSCHMIDT A.-G. (G.P. 423,292, 8.6.23. Addn. to 420,068; B., 1926, 331).—A bronze-base bearing metal contains 14—16% Pb, 2·5—3·5% Ni, 2—6·5% Sn, and the remainder copper. Substitution of antimony and zinc for part of the tin increases the hardness, compression strength, plastic and bearing properties. A. R. POWELL.

Aluminium solder. J. DE MAY, Assr. to PACKARD MOTOR CAR Co. (U.S.P. 1,584,219, 11.5.26. Appl., 26.2.19).—A solder for aluminium comprises over 50% Sn with a large percentage of zinc and substantial amounts of aluminium and antimony. A. R. POWELL.

Heat treatment of oxidised copper ores. J. C. MOULDEN, B. TAPLIN, and METALS PRODUCTION, LTD. (E.P. 250,991, 29.10 and 27.11.24, and 3.4.25).—Refractory oxidised copper ores, *e.g.*, ores containing chrysocolla, are mixed with a small proportion of sodium chloride and with sufficient carbonaceous matter to effect complete reduction of the copper. The mixture is fed into a rotating furnace, in which a neutral atmosphere is maintained, and heated therein to a temperature above 500°, whereby the copper compounds are decomposed and the copper is deposited on particles of carbonaceous matter, building up small balls of the metal which, after cooling the charge to avoid oxidation, may be separated by screening or gravity concentration or by leaching with ammoniacal solutions of ammonium carbonate. Should the ore contain alkaline material, this must be removed by leaching with water or by neutralisation with sulphuric acid prior to the furnace treatment. A. R. POWELL.

Electrodeposition of nickel. E. A. OLLARD, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (E.P. 251,010, 21.1.25).—In order to obtain smooth electrodeposits of nickel free from pitting, the bath is kept in agitation during electrolysis by means of a current of ozonised air or oxygen. Alternatively the bath may be saturated with ozone from a stream of gas containing it, previous to use. A. R. POWELL.

Galvanising and similarly coating metals. J. B. HILL (E.P. 251,197, 26.10.25).—In galvanising or lead-coating steel or sheet iron, the metal as it comes from the coating bath or from the exit rollers attached to the bath is sprayed with a mixture of ammonium chloride, ammonium sulphate, or rosin, and sulphur. The mixture may be applied as a liquid or in the form of fine dust carried by a current of air or inert gas under pressure. The surface of the resulting sheets is highly lustrous and resembles tinplate. A. R. POWELL.

Electrodeposition of metals. C. P. MADSEN, Assr. to MADSENELL CORP. (U.S.P. 1,583,891, 4.5.26. Appl., 19.9.18. Renewed 8.10.25).—In order to remove the hydrogen from an electrodeposited metal the cathode is periodically removed from the bath for a time sufficient to allow of the elimination of hydrogen bubbles from its surface. A. R. POWELL.

Treatment of ores for the recovery of titanium, tungsten, and tantalum. A. W. GREGORY (E.P.

251,527, 20.10.25).—In order to separate the iron from ilmenite, wolframite, or tantalite, the powdered mineral is mixed with a small proportion (*e.g.*, 20%) of sodium carbonate and with sufficient carbon just to reduce the iron compounds to metal. The mixture is heated to 700—800° in a neutral or reducing atmosphere for some time, and the reaction product is extracted with dilute sulphuric acid, which dissolves the sodium salts and the metallic iron formed, leaving a residue of titania, tungsten oxide, or tantalum oxide for further purification. A. R. Powell.

Lead refining. H. M. Schleicher, Assr. to Amer. Metal Co. (U.S.P. 1,583,495, 4.5.26. Appl., 6.11.25).—Lead containing impurities is added to a bath of pure molten lead maintained in contact with an oxidising agent and a reagent capable of absorbing the oxidised impurities. The rate of addition of crude lead is controlled by the rate of absorption of the oxidised impurities by the reagent. L. M. Clark.

Separating metal from material containing it. E. B. Kirby (U.S.P. 1,583,933, 11.5.26. Appl., 11.8.23).—The material is smelted in a blast furnace with fluxes to make a very viscous slag at the temperature at which the metal volatilises and with sufficient fuel to vaporise the whole of the metal. The charge is first heated at a temperature at which the metal compounds are reduced and then passes into the hot zone of the furnace from which the waste gases and metal vapour are removed, the bulk of the metal being condensed to a liquid state in a condenser maintained at a temperature above the m.p. of the metal, and the remaining metal, together with the waste gases being passed back into the furnace at a point higher than that at which it was removed. A. R. Powell.

[Preparing and] volatilising metal [magnesium]. H. E. Bakken, Assr. to Amer. Magnesium Corp. (U.S.P. 1,584,689, 11.5.26. Appl., 27.4.21).—Magnesium is prepared by electrolysing its fused salts and simultaneously volatilising the metal under reduced pressure. B. M. Venables.

Metallurgical apparatus [shaft furnace]. W. E. Greenawalt (U.S.P. 1,585,344, 18.5.26. Appl. 19.8.24).—An electrically-heated vertical shaft furnace consists of two sections the upper of which serves for heating the charge and tapers upwards towards the charging hopper, and the lower serves as a cooling chamber and tapers downwards towards the discharge outlet from which the reaction product is removed continuously. Heating in the top section is effected by means of a series of electrodes at varying elevations, and means are provided at the junction of the two sections for injecting a fluid into the reacting mass. A. R. Powell.

Purification of molybdenite containing copper and bismuth compounds. E. Pokorny (G.P. 426,431, 14.7.23. Addn. to 425,364; B., 1926, 496). —The ore mixture is subjected to oil flotation in an alkaline pulp through which chlorine is passed. A. R. Powell.

Treatment of aluminium, magnesium, their alloys and other metals, to render them impervious to fluids, and to inhibit corrosion. S. Daniels, A. C. Zimmerman, and J. A. Watson (E.P. 252,070, 16.2.25).—See U.S.P. 1,540,766; B., 1925, 677.

Magnetic [ore] separators. H. S. Hatfield (E.P. 252,034, 8.12.24).

Apparatus for treating liquid with gas (U.S.P. 1,583,141 and 1,583,591).—See I.

Enamelling metal (E.P. 251,327).—See VIII.

XI.—ELECTROTECHNICS.

See A., June, 583, **Electrolytic deposition of antimony from acid and alkaline solutions** (Lukas and Jilek); **Anodic oxidation of chromic oxide** (Saxon).

Patents.

Lead accumulators. Soc. Anon. le Carbone (E.P. 235,530, 20.4.25. Conv., 14.6.24).—The weight of lead accumulators may be reduced and their specific capacity increased by using, as the active material, very finely powdered lead or an oxide of lead, mixed with a porous conductor such as coarse-grained charcoal powder, which has been rendered impermeable to liquids by mixing with very fine-grained charcoal powder. Such a porous conductor absorbs, during charging, oxygen and hydrogen which have not been fixed by the lead and lead oxide respectively and evolves these gases on discharge; it also retards the corrosion of the lead. M. E. Nottage.

Lead accumulators. Soc. Anon. le Carbone (E.P. 244,399, 21.4.25. Conv., 11.12.24. Addn. to 235,530, preceding).—The porous grains of the active material may be rendered impermeable to liquids by exposing them to the vapours of fatty substances, or by moistening them with a solution of fatty substances or of rubber, the solvent being subsequently evaporated. The grains are then mixed with powdered lead or an oxide of lead, and moistened with sulphuric acid. The active material so formed is mixed with an agglomerate or binder, and compressed in a mould having grooves or ribs on its surface. It is heated to expel the liquid of the binder, and molten lead is then poured into the recesses of the plate. Accumulator plates made in this way are not liable to disaggregation at high charging and discharging rates. M. E. Nottage.

Heating liquids [electrically]. Österreichische Chem. Werke G.m.b.H. (E.P. 242,253, 9.10.25. Conv., 28.10.24).—In apparatus for heating liquids electrically, in which the liquid is itself used as resistance, the liquid passes through distributing structures provided with interstitial spaces in such manner that these spaces are filled only partly, so that no hydrostatic pressure preventing free escape of the gases or vapours generated by the heater, is produced. For example, the liquid may

trickle over and through columns of porous material arranged between electrodes. J. S. G. THOMAS.

Electrolysis or the splitting up of air or water. T. D. KELLY (E.P. 251,021, 23.1.25).—Prior to electrolysis, the air or water is made electrically conducting by ionisation by means of radioactive light transmitted through quartz or other media transparent to the rays. By this means large quantities of nitrogen, oxygen, and hydrogen may be obtained with a small expenditure of electricity.
M. E. NOTTAGE.

Electrolysis of alkali chlorides with horizontal diaphragms. ZELLSTOFF-FABR. WALDHOF, and R. TAUSSIG (G.P. 427,085, 16.8.22).—Alkali chlorides are electrolysed in horizontal cells between cooled cathodes and graphite anodes, the surfaces of which are increased by the provision of channels and prominences produced by burning off tar from a portion of the surface and subsequently graphitising the residual carbon. A. R. POWELL.

***X*-Ray apparatus.** W. MOPPETT (E.P. 251,477, 9.7.25).—An *X*-ray tube having a uranium anticathode and used in conjunction with one or more screens, of which at least one contains uranium, is used for generating and controlling monochromatic *X*-ray *K*-radiations in biological, therapeutic, or other operations. J. S. G. THOMAS.

Electric furnaces. E. C. R. MARKS. From SCOVILL MANUF. CO. (E.P. 251,484, 13.7.25).—In an electric furnace in which current is supplied to an electrode by a transformer, the repulsive force developed between the primary and secondary elements of the transformer controls the position of the electrode with respect to the charge.
J. S. G. THOMAS.

Nitric oxide by electrical methods. J. S. ISLAND (F.P. 601,740, 8.8.25).—The electrodes are connected to an industrial alternating current network and supplied with a high-tension auxiliary current, the auxiliary circuit forming a branch of the network of the main current and being provided with an arrangement for the production of a current of high frequency. A. COULTHARD.

Electric furnaces. G. EVREYNOFF (E.P. 252,037, 11.12.24).—See U.S.P. 1,562,825; B., 1926, 134.

Electrodeposition of nickel (E.P. 251,010).—See X.

Electrodeposition of metals (U.S.P. 1,583,891). —See X.

Magnesium (U.S.P. 1,584,689).—See X.

Electrodeposition of rubber (U.S.P. 1,583,704). —See XIV.

XII.—FATS; OILS; WAXES.

Detection of olive oil obtained by extraction with solvents. S. FACHINI (Giorn. Chim. Ind. Appl., 1926, 8, 178—179).—The presence in expressed olive oil of even a small proportion of oil either extracted with solvents or extracted from the pressed residue and then refined, may be detected by the following reaction, analogous to Morawski's reaction for resin. A mixture of 2—3 c.c. of the oil with an equal volume of acetic anhydride is heated in a test-tube and shaken for a short time, cooled, and filtered through a small filter wetted with acetic anhydride. In contact with a few drops of concentrated sulphuric acid in a porcelain dish, a little of the filtrate yields a cherry-red coloration if extracted oil is present. If this liquid is then treated with a few c.c. of water, a more or less intense green coloration appears and subsequently gradually disappears. T. H. POPE.

Constituents of crude cottonseed oil. G. S. JAMIESON and W. F. BAUGHMAN (J. Oil Fat Ind., 1925, 1, 30—34; Chem. Abstr., 1926, 20, 1724).—The acetone-insoluble portion of settlings of crude cottonseed oil contained SiO_2 0·60, P_2O_5 6·14, CaO 0·26, MgO 1·46, K_2O 1·79, Na_2O 0·33%. The phosphorus is present partly as phosphate and partly as a phosphatide related to vegetable lecithins (*cadmium chloride* compound, m.p. 194—196°). Almost all of the phosphorus compounds are removed when the crude oil is refined with sodium hydroxide.
A. A. ELDRIDGE.

Micro-reaction for cottonseed oil. J. PRESCHER (Z. Unters. Lebensm., 1926, 51, 234—235). —The Halphen reaction can be carried out in a capillary U-tube as used for m.p. determinations, a distinctly recognisable coloration being obtained with minute quantities of cottonseed oil.
B. W. CLARKE.

Seed and oil of *Johannesia Princeps*. G. ETZEL and C. G. KING (J. Amer. Chem. Soc., 1926, 48, 1369—1372).—Crushed kernels of nuts of *Johannesia Princeps* yielded on extraction with ether, 55·7% of oil, whilst cold pressing gave a yield of 26·6%. The oil is pale yellowish-green and clear, not disagreeable to the taste, $d^{15.5}$ 0·9257, d^{20} 0·9229, n^{15} 1·4770, n^{20} 1·4750, iodine value (Hanus) 115·67, saponif. value 192·15, acid value 5·44, acetyl value, 8·75, Reichert-Meissl value 1·2, Polenske value 0·345, and unsaponifiable matter 1·17%, from which a phytosterol, m.p. 131°, was isolated. Examination of the acids by the Gusseroff-Varrentrap method indicated that these contained 80·45% of unsaturated and 7·79% of saturated acids (cf. Niederstadt and Peckol, Ber. Pharm. Ges., 1905, 15, 225). The oil is semi-drying, and is a useful cathartic (cf. New Remedies, 1881, 10, 260; Pharm. J. Trans., 1881—1882, 41, 380; Monit. Pharm., 1881, 156). The press-cake contains 10·05% of nitrogen and 13·29% of phytin, and should form a valuable fertiliser.
F. G. WILLSON.

Wild duck oil. HIROSE (J. Soc. Chem. Ind. Japan, 1926, 29, 17—19).—An oil obtained from the skin and flesh of a wild duck had d_4^{30} 0·9097, saponif. value 198·9, iodine value 81·6, n^{40} 1·4619, acid value 2·2, and unsaponifiable matter 0·41%. The mixed fatty acids had iodine value 83·4, n^{40} 1·4520, neutralisation value 205·4, and mean mol. wt, 273·2. The

liquid and solid acids were separated by the lead salt-ether method and amounted to 71% and 29% respectively. The liquid acids had iodine value 103·5, n^{40} 1·4550, and neutralisation value 202·0. The solid acids had iodine value 25·1, neutralisation value 213·6, and m.p. 54·5—56·0°. The liquid acids gave 21·47% of tetrabromolinoleic acid; other highly unsaturated acids were not present. The above data and the distillation test of the methyl esters of the acids show that the unsaturated fatty acids of the oil are composed of oleic and linoleic acids and the saturated acids of stearic and palmitic acids, and possibly myristic acid. K. Kashima.

Standardisation of drying oils. Group of paint oils containing linolenic acid. Conifer seed oils and the isomeric linolenic acids. A. Eibner and F. Reitter (Chem. Umschau., 1926, **33**, 114—129).—An investigation of spruce and fir seed oils shows that while possessing suitable properties (especially rapidity of bleaching) for use in artists' colours, they are of little value for paints in general, since they dry slowly and yield dark varnishes, the films of which readily soften under heat. The presence of β-linolenic acid in exceptional quantity places these oils in an intermediate group between linseed oil and the walnut-poppy seed oil group. β-Linolenic acid yields a tetrabromide only and is richer in energy albeit poorer in drying power than the hexabromide-yielding α-isomer which constitutes 20% of linseed oil. This renders compatible the conflicting iodine and hexabromide values (172 and 14 respectively) of fir seed (*Pinus sylvestris*) oil which was found to contain 6·67% of α-linolenic acid, 17·28% of β-linolenic acid, 30·82% of α-linoleic acid, 23·94% of β-linoleic acid, 9·01% of oleic acid, 6·80% of saturated acids, 1·07% of unsaponifiable matter, and 4·26% of glycerol residue. Spruce seed oil contains 5·20% of α-linolenic acid, 15·67% of β-linolenic acid, 29·55% of α-linoleic acid, 23·60% of β-linoleic acid, 11·43% of oleic acid, 7·64% of saturated acids, 1·00% of unsaponifiable matter, 1·46% of hydroxy-acids, and 4·61% of glycerol residue. These figures tabulated alongside analyses of linseed, walnut, and poppyseed oils (due to Schmidinger, Wick, and Wibelitz respectively) clearly show the gradation that exists, and explain to some extent the difficulty of recognising the mixed glycerides in the conifer seed oils. Only very small quantities of these oils can be obtained at present.

S. S. Woolf.

Fatty acids of Aizame liver oil. Fatty acids of shark and ray liver oils. II. M. Tsujimoto (J. Soc. Chem. Ind. Japan, 1926, **29**, 67—71).—The fatty acids from a sample of "Aka-aizame" (*Centrophorus lusitanicus*) liver oil contained about 10% of saturated acids, which consisted chiefly of palmitic acid with a small amount of selachoceric acid, $C_{24}H_{48}O_2$; stearic acid was present only in a far smaller amount, if at all. The amount of unsaturated acids was about 90%. They consisted essentially of acids of the oleic series, viz., oleic acid, an acid $C_{20}H_{38}O_2$ (gadoleic acid ?), an acid $C_{22}H_{42}O_2$ (an isomer of erucic acid, probably identical with the cetoleic acid of Toyama), and selacholeic acid, $C_{24}H_{46}O_2$; an acid $C_{16}H_{30}O_2$, was also probably present. Unsaturated acids of the $C_nH_{2n-4}O_2$ series and more highly unsaturated acids were present in small amount. K. Kashima.

Relations between fat constants. G. Schay (Z. angew. Chem., 1926, **39**, 729—731).—Assuming the additivity of molecular volume and molecular refractivity the following equations are deduced for fats: $d=\alpha[1+\beta(1+\gamma I)N]$ and $n=a+bN+cI$, where d is the density, n the refractive index, N the saponification value, I the iodine value of the fat, and α, β, γ, a, b, and c are constants. Lund's relationships (B., 1922, 944 a) immediately follow from these equations as first approximations (cf. following abstract). S. K. Tweedy.

Refraction of brominated fats. A. Schwicker and G. Schay (Z. angew. Chem., 1926, **39**, 731—732).—The saponification and (Winkler) bromine and iodine values of some fats are recorded together with their refractive indices, $n_D{}^{40}$, and those of their bromo-derivatives. The relationships deduced for iodine values (preceding abstract) also hold for bromine values. A formula involving n is also deduced for the brominated fats. Becker's method (B., 1923, 1185 a) gave bromine values which were always too high. S. K. Tweedy.

Significance of liquid-liquid interfacial tension in the qualitative detection of unsaponifiable oils in fats. D. Holde and A. Gorgas (Chem. Umschau, 1926, **33**, 109—113).—The formation of recognisable emulsions when 6—8 drops of oil are boiled for 2 min. with 2½ c.c. of N-alcoholic potash and then mixed with water, constitutes a standard qualitative test for unsaponifiable matter in saponifiable oils and fats, but owing to low interfacial tension between the soap solution and the unsaponifiable matter, appreciable amounts of the latter escape detection. The drop numbers of a petroleum product ("DEA") against linseed, poppyseed, cottonseed, and bone oils at 15° and 75° show that the greatest interfacial tension is exhibited in linseed oil at the higher temperature. From the results of these experiments, three modifications of the qualitative test are suggested, viz., addition of hot instead of cold water; cooling the soap solution before addition of water; and addition of linseed oil and glycerol to the soap solution. Each of these modifications renders the test more delicate, by the raising of interfacial tension. S. S. Woolf.

Purification of glycerin water. O. Hausamann (Chem.-Ztg., 1926, **50**, 369—371).—The purification of glycerin water from autoclave saponification is effected by (1) free blowing off of the autoclave during saponification, (2) treatment with milk of lime after a preliminary concentration to d 1·11, (3) treatment of the filtered water from the latter operation with barium hydroxide, and (4) treatment with oxalic acid to remove barium hydroxide. The evaporation of the final filter-press water is said to give a product yielding an ash not exceeding 0·2%, and organic residue not exceeding 1·0%.

If the water is to be subsequently used for the manufacture of "C.P." glycerin, after the liming treatment it is filtered with animal charcoal, which, in addition to decolorising the lyes, is also stated to retain some of the volatile acids. A. RAYNER.

See also A., June, 571, **Fat solvents** (BILLS). 596, **Synthesis of waxes** (GRÜN, ULBRICH, and KREZIL). 597, **Preparation of stearolactone** (BLUMENSTOCK). 638, **Biological significance of unsaponifiable matter of oils** (CHANNON; CHANNON and MARRIAN).

Oxidation of petroleum oils. PETROV and DANILOVICH.—See II.

PATENTS.

Recovery of oils from the fat-bearing parts of marine animals. K. HOLTER and S. THUNE (E.P. 232,954, 21.4.25. Conv., 22.4.24).—The material is boiled with water, and, after straining off the coarser impurities, the fat is separated from the aqueous liquor by centrifuging at 60—100°. L. A. COLES.

Deacidification, deodorisation and clarification of oils, fats and waxes. E. FORAY (F.P. 600,948, 20.7.25).—The oil or melted fat or wax is mixed with powdered, porous charcoal, for example, wood charcoal, the mixture heated to 100—150°, and the charcoal filtered off. The charcoal may be mixed with 1% of a catalyst. A. COULTHARD.

Apparatus for the distillation of oils and fats. CONTINENTALE A.-G. FÜR CHEMIE U. REICHSVERKEHRSBANKE (G.P. 426,233, 12.12.24).—Three vacuum vessels, are used, the first and second serving for distillation of the material with superheated steam, and the third for drawing off the distillation product from the second vessel and cooling it to the normal temperature or to a temperature at which oxidation is excluded. A cataract condenser and an air pump are connected with the third vacuum vessel to remove the more volatile constituents. The formation of emulsions and darkening of the distillate are avoided. A. COULTHARD.

Non-odorous phenolated soap. N. SULZBERGER (U.S.P. 1,585,434, 18.5.26. Appl., 15.3.23; cf. U.S.P. 938,614, B., 1909, 1273 A).—Antiseptic soaps are prepared by mixing with ordinary soaps about 3% of an ester formed from a phenol and a fatty acid with more than 11 carbon atoms in the molecule, such as phenyl stearate, phenyl palmitate, or phenyl oleate. These esters are non-irritant, have no odour of phenol, and are stable to the alkali present in the soap. T. S. WHEELER.

Purification of soap solutions. H. HARRIES, Assee. of C. HARRIES (G.P. 424,409, 23.8.22).—The lathering properties of soap solutions are improved by subjecting the solution, heated to facilitate separation of oil, to the action of an electric current. L. A. COLES.

Solvent for fats (G.P. 425,511).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Flash-points of paints and varnishes. J. C. SMITH and F. B. CROW (J. Oil and Colour Chem. Assoc., 1926, 9, 112—116).—A series of pastes, paints, and varnishes containing varying amounts of volatile solvent, were found in every case to have higher flash-points than the white [petroleum] spirit or turpentine used, even when the solvent was present to the extent of 25%. The flash point rose with decrease in amount of thinner. Using three samples of white spirit (flash-points 24·4°, 26·7°, and 35° respectively) it was found that the mean rise of flash-point in corresponding mixtures was independent of the flash-point of the particular white spirit used. While the flash-points of mixtures containing turpentine followed the same trend as those containing white spirit, no relationship could be deduced, as the rise in flash-point of turpentine mixtures was in one series more and in another less than the rise of flash-point of corresponding white spirit mixtures. S. S. WOOLF.

Crystalline glyceryl triabietate and abietic anhydride. E. FONROBERT and F. PALLAUF (Farben-Ztg., 1926, 31, 1848-1849).—Pure abietic acid was obtained from rosin (colophony) by cooling a 50% solution in acetone, which was found to be a superior solvent to benzene and ethyl acetate both from the point of view of yield and of purity of product; 1000 g. of rosin gave 520 g. of abietic acid, which after a further recrystallisation had m.p. 165°, acid value 183·7. A pure rosin-glyceride was dissolved in an equal weight of hot acetone and the crystals formed on cooling were washed with acetone and dried; the yield of glyceryl triabietate exceeded 10%. This was repeated with glyceryl esters synthesised from samples of French, Spanish, American, and German rosin, and with an English ester-gum, and yields of crystalline glycerides of 3—7% were obtained. The product was insoluble in ethyl alcohol, thus differing from the readily soluble mono- and di-glycerides (which were synthesised from mono- and di-chlorohydrin) and is identical with the crystalline ester obtained by Wolff (cf. B., 1926, 501). Pure abietic acid was obtained by saponification of this ester. Pure abietic acid dehydrated with acetic anhydride is sparingly soluble in acetone and ether, but may be re-crystallised from benzene, ethyl acetate, and light petroleum, the last-named giving the purest product. The crystalline anhydride, obtained in more than 50% yield from light petroleum solution, has m.p. 151°. S. S. WOOLF.

Turpentine from *Picea excelsa*, Link. A. VELCULESCU (Bul. Chim. Soc. Româna Stiinte, 1925, 28, 29—33).—The clear yellow to dark chestnut coloured exudation from *Picea excelsa* yields, on steam distillation, 4—9% of volatile oil having b.p. 154—185°, d^{20} 0·864+0·869, n^{20} 1·473—1·477, $[\alpha]_D^{20}$ —11 or —12°; 44% of the distillate boils at 165—170° and is probably β-pinene. The resin obtained by extracting the non-volatile residue with alcohol forms 40—60% of the original mass. It is a dark

brown solid of m.p. 68—72°, d 1·072—1·083, acid value 113—133, saponification value 136—150, ester value 10—23 (cf. B., 1924, 479). H. E. F. Notton.

Chemical composition of rosin. D. N. Shaw and L. B. Sebrell (Ind. Eng. Chem., 1926, 18, 612—614).—Samples of abietic acid prepared from American rosin by five different methods had different physical characteristics but were all of the same composition, $C_{20}H_{30}O_2$. In the complete absence of free oxygen, abietic acid is not easily dehydrated; rosin is therefore probably essentially abietic acid and not an anhydride. The readiness with which abietic acid undergoes oxidation to a mono-hydroxy-derivative which when heated readily yields an anhydride probably explains the discrepancies between earlier results (cf. Knecht and Hibbert, B., 1919, 472 A; Knecht, B., 1923, 1234 A; Ruzicka and Schinz, B., 1923, 800 A). D. F. Twiss.

Condensation products of phenols and aldehydes. II. Colour reactions of the products formed by using ammonia as a catalyst. T. Shono (J. Soc. Chem. Ind. Japan, 1926, 29, 53—58).—Alcoholic solutions (5%) of the phenol-formaldehyde condensation products formed by using ammonia and hydrochloric acid separately as catalysts give colour reactions with alcoholic solutions (1%) of metal salts. These colour reactions are very distinct and can be applied for the identification of the resins. The reactions with 10 common natural resins are given for comparison. Among the compounds used, the following give the most characteristic colour reactions: chloroauric acid, chloroplatinic acid, uranium nitrate, and chlorides of cobalt, iron, vanadium, selenium, titanium, ruthenium, tungsten, etc. The phenol-formaldehyde condensation product formed by using ammonia as a catalyst and soluble in alcohol, even after repeated purification by solution in alcohol and re-precipitation, still contained nitrogen when tested by Lassaigne's method. K. Kashima.

Detection of resins, particularly in linseed oil varnishes. K. Brauer (Chem.-Ztg., 1926, 50, 371—372).—Linseed oil gives the Liebermann-Morawski reaction with acetic anhydride and sulphuric acid, and thus this test cannot be used as a means of detecting resins. A general reaction for resins is given by phosphomolybdic acid in presence of ammonia, in ethereal solution, a blue colour being produced. By this means resins may be distinguished from linseed oil, which gives a pale green colour under these conditions. Diazotised sulphanilic acid also gives orange or red dyes with all resins owing to the phenolic nature of these compounds. To distinguish between shellac and colophony the sample is treated with 0·1 to 0·2 g. of finely powdered ammonium molybdate dissolved in 5 c.c. of concentrated sulphuric acid; this reagent gives with shellac a green, and with colophony a lilac colour. Phosphotungstic acid in presence of ammonia with colophony gives a green colour, and with shellac a lilac colour. Tables are given showing the colours developed by various resins with the above reagents and also with sulphuric acid. A. Rayner.

See also A., June, 558, **Phosphorescent sulphides of zinc** (Guntz). 588, **Oranges and reds of uranium** (Auger and Longinescu). 611, **Crystalline salts of abietic acid** (Dupont, Desalbres, and Bernette; Dupont and Desalbres).

Paint oils containing linolenic acid. Eibner and Reitter.—See XII.

Patents.

Bituminous paints and like coating and impregnating compositions. G. S. Hay (E.P. 251,323, 1.1.25).—Aqueous bitumen emulsions break down on agitation with bitumen solvents (*e.g.* naphtha, kerosene, benzine, trichloroethylene) or inorganic substances (*e.g.* calcium chloride, common salt, caustic soda, potassium iodide). Further addition of bitumen solvents and more rapid agitation effect a re-emulsification with probable reversal from the original bitumen-in-water emulsion to a water-in-bitumen emulsion. The water-in-bitumen emulsions adhere readily to metallic surfaces (in contradistinction to the simple bitumen-in-water type) and may also be used for impregnating paper or fabric for waterproofing purposes. S. S. Woolf.

Drying lithopone. Gewerkschaft Sachtleben (E.P. 251,502, 1.9.25. Conv., 30.7.25).—Precipitated lithopone from the filter-press, travels through a rotary furnace in the same direction as hot, waste, non-oxidising gases (freed from carbon dioxide, dust, etc.), the drying temperature being controlled. As the compressed cakes are broken down in this process, subsequent grinding for the final calcination is avoided or facilitated. S. S. Woolf.

Calcining crude lithopone and other materials. A. S. Krebs, Assr. to Krebs Pigment & Chemical Co. (U.S.P. 1,584,381, 11.5.26. Appl., 14.4.23).—The material is treated in a rotary muffle tube, a portion of the tube at the inlet end being heated externally, whilst the remainder is not heated, but is insulated against loss of heat. B. M. Venables.

Grading materials [carbon black]. H. W. Price, Assr. to J. M. Huber Co. (U.S.P. 1,585,413, 18.5.26. Appl., 21.5.24).—Carbon black is refined by agitation so as to cause air flotation of a portion, while other portions are simultaneously passed through a sieve, and undesirable particles are removed by impinging them upon a bed of the unagitated material. S. S. Woolf.

Manufacture of a pigment from galena. A. E. Morgans (Austral. P. 18,759, 21.7.24).—A mixture of galena and fuel is heated to redness in a furnace open at the upper end and a current of air is driven downwards through the glowing mass. A. R. Powell.

Production of material [for use in the manufacture of paints] from the fruit of *Rhamnus* species. Maison Breton, J. Fichot & Cie., and

G. L. A. CRUT (F.P. 598,078, 21.8.24).—Rhamnetin obtained by boiling with acids extracts of *Rhamnus infectoria* or *R. cathartica*, can be used for the production of lakes with the aid of alumina or stannic oxide, with or without the addition of mordants.

L. A. COLES.

Production of black ink [from nitro-dinaphthylene dioxide] resistant to the action of acids and water. J. INOUYE (F.P. 600,390, 7.7.25).—Solutions suitable for use as ink are prepared by dissolving tetra– or hexa-nitro-derivatives of dinaphthylene dioxide, preferably after sulphonation, in dilute ammonia. For example, dinaphthylene dioxide, obtained by heating β-naphthol with cupric oxide or manganese dioxide under pressure, is converted by heating with nitric acid, *d* 1·38, for 2—5 hrs. at 100° into its tetranitro-derivative or for 50 hrs. at 110° into its hexanitro-derivative, and the product is sulphonated and dissolved in ammonia.

L. A. COLES.

Recovering by-products from plant substances etc. F. K. FISH, JUN. (U.S.P. 1,574,614, 23.2.26. Appl., 2.1.20).—Resinous woods or plant substances are treated under pressure with superheated water charged with the volatile constituents of wood (gums, terpenes, etc.) liberated in a similar, previous process. To facilitate extraction of the volatile substances the pressure is decreased periodically by spraying the steam with cold water. Water and liberated substances are afterwards separated.

R. B. CLARKE.

Colourless compounds containing sulphur and suitable for adsorption on textile fibres and other substrata. A.-G. F. ANILIN-FABR. (E.P. 242,974, 4.11.25. Conv., 14.11.24. Addn. to 232,958 ; cf. U.S.P. 1,553,014, B., 1925, 916).—The product of reaction of hydroxybenzenes and sulphur chloride is treated with concentrated sulphuric acid and excess of the hydroxybenzene at a high temperature. The products thus obtained are soluble in aqueous sodium carbonate and are precipitated by acids as colourless flakes. The lakes formed with basic dyestuffs are faster to light than those formed by the substances prepared according to the original patent.

A. GEAKE.

Magnesium-containing synthetic resin. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,580,424. 13.4.26. Appl., 11.6.23).—Formaldehyde and phenol are heated with 0·25—0·5 mol. of magnesium hydroxide to give a solution which on evaporation yields a strong resinous product capable of being mixed with the usual organic and inorganic fillers, with the exception of silicates. The yield of resin is 120% of the weight of phenol taken.

E. S. KREIS.

Moulding composition containing organo-magnesium compounds. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,580,425, 13.4.26. Appl., 25.6.23).—By heating together phenol, magnesium hydroxide, and 40% formaldehyde, a solution is obtained which yields 56% of solid material. An equal weight of wood flour or other filler can be added to the solution and the excess moisture removed *in vacuo*. Under a vacuum of 28 in. of mercury, the heating should be discontinued when the residue reaches a temperature of 90°. The composition is cured in a hot press at 160—170°.

E. S. KREIS.

Furfuraldehyde–acetone resins. L. T. RICHARDSON, Assr. to CUTLER-HAMMER MFG. Co. (U.S.P. 1,584,144, 11.5.26. Appl., 10.8.25).—Resins are obtained by interaction of furfuraldehyde and acetone in the successive presence of alkalis and acids.

L. A. COLES.

Manufacture of artificial resins. A. REGAL (U.S.P. 1,584,472–3, 11.5.26. Appl., 17.6.25).—Formaldehyde is heated with phenols in the presence of (A) an indophenol, formed by adding to the mixture a small quantity of a *p*-aminoaryl-compound, followed by moderate oxidation, or (B) additive products formed by the action of formaldehyde on derivatives of aromatic amines in which the amino-hydrogen atoms are replaced by organic radicals.

L. A. COLES.

Manufacture of condensation products from phenols or their derivatives and acetaldehyde. CHEM. FABR. GÜSTROW, HILLRINGHAUS & HEILMANN (G.P. 422,904, 21.2.14 ; cf. E.P. 163,679 ; B., 1922, 261 A).—The formation of acetaldehyde and of phenol-acetaldehyde condensation products can be combined by leading acetylene into a mixture of a phenol and a strong acid (or a phenolsulphonic acid) in the presence of mercury salts in solution. For example, crude phenol or cresol is added to a solution of mercuric oxide in 30% sulphuric acid and acetylene passed into the vigorously stirred mixture. When the absorption is complete, the viscous reaction product is separated from the aqueous solution, washed, and the excess phenol driven off by steam. After removing the mercury (wholly or partly in combination) by treatment with alcohol or benzene, the transparent resinous condensation product, m.p. about 100° (after previous softening) is soluble in sodium and potassium hydroxides but not in their carbonates. Other acids such as hydrochloric, hydrofluoric, phosphoric, or acetic acid, may be used instead of sulphuric, and sulphonic acids of phenols instead of phenols, in which case the addition of inorganic acid is unnecessary or even harmful. On using phenolsulphonic acid alone, the condensation products are soluble in water ; using a mixture of phenol and its sulphonic acid, the resin insoluble in water is also obtained. The products are used as substitutes for natural resins or as tanning materials.

A. COULTHARD.

Manufacture of derivatives of condensation products of phenols containing sulphur. GES. FÜR CHEM. IND. IN BASEL (G.P. 425,798, 7.8.24. Conv., 31.10.23. Addn. to 418,498, cf. E.P. 186,107 and 203,310, B., 1922, 905 A ; 1924, 566).—Resin esters, similar to those of the chief patent, are obtained by treating phenols or naphthols with

sulphur halides and acylating reagents in the presence (if necessary) of water, aqueous alkalis or other solvents and diluents. Examples are given of the preparation of resin esters from phenol and acetyl chloride or acetic anhydride, phenol and benzoyl chloride, tricresol and acetic anhydride, a mixture of phenol and tricresol and benzoyl chloride, tricresol and benzoyl chloride, resorcinol and benzoyl chloride, α-naphthol and acetic anhydride, and α-naphthol and benzoyl chloride, together with sulphur chloride in each case. When hydrogen chloride ceases to be evolved, the last traces of acid and other volatile substances are removed in a vacuum, or the resin is washed with water until neutral. Harder resins are obtained when the amounts of sulphur chloride used are greater. A. COULTHARD.

Improving the quality of resins. CONSORTIUM FÜR ELEKTROCHEM. IND. G.M.B.H., Assees. of E. BAUM and W. O. HERRMANN (G.P. 426,283, 21.2.22). —Resin is treated with chlorine or with hypochlorous acid, and the product treated with aqueous alkaline solutions until the combined chlorine is removed. The water-resisting properties of the product are improved by precipitating it from solution in a suitable solvent, or by treating it with water or, preferably, with dilute acids. L. A. COLES.

Artificial ageing of amber. E. SAXL (Austr. P. 101,950, 29.1.24).—Amber is treated with steam under pressure in the presence of catalysts such as acids, bases, or salts. For example, it is treated with 70—90% alcohol, and then with 4—10% hydrochloric acid under pressure at above 100°. L. A. COLES.

Solvent for extracting resins and fats. J. D. RIEDEL A.-G. (G.P. 425,511, 25.1.25. Addn. to 320,807; B., 1920, 665 A).—The use of chlorinated tetrahydronaphthalene is claimed. L. A. COLES.

Ferric oxide (E.P. 251,310).—See VII.

Oxidised leaden powder [litharge] (E.P. 251,449).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Separation of caoutchouc hydrocarbon from rubber latex and its fractionation. R. PUMMERER (Kautschuk, 1926, April, 85—88).—Hevea latex (750 g. of approximately 40% concentration), preserved with ammonia, is mixed with sodium hydroxide solution (750 g. of 8% concentration) and water (1250 c.c.) for 8 hrs. at 50°. This mixture, when kept, yields a "cream" which is separated and further purified by two repetitions of the treatment with dilute sodium hydroxide. The cream is then washed with water and purification is finally completed by dialysis in an atmosphere of nitrogen. After coagulation with acetone, the product, which is resin-free, is dehydrated by extraction with acetone in a Soxhlet apparatus and then dried under reduced pressure at ordinary temperature. The product is a transparent, pale yellow mass which is not sticky, contains no nitrogen, and gives only 0·077% of ash (cf. de Vries and Beumée-Nieuwland, B., 1925, 891). By repeated extraction with ether at room temperature it is resolvable into a soluble "ether-sol-caoutchouc" (65—75%) and an insoluble "ether-gel-caoutchouc," both of the empirical composition C_5H_8. The gel product always yields further quantities of the soluble form on further treatment with ether. D. F. TWISS.

Condition of the "resin" in rubber. H. LOEWEN (KAUTSCHUK, 1926, April, 90—91; cf. Reiner, B., 1926, 288).—A critical review of the experimental method of Reiner and others, shows that there is no evidence of any form of attachment of the resins to rubber either chemically or by adsorption, the resins probably being merely in a state of solution. D. F. TWISS.

Effect of heat, generated during stressing, on the tensile properties of rubber. C. E. BOONE and J. R. NEWMAN (Ind. Eng. Chem., 1926, 18, 539—540).—Rubber generates heat so rapidly, when stressed, *e.g.*, under the ordinary conditions of testing, that its temperature may rise from 6° to 12° above that of its surroundings. Cooling by a fan during testing causes the observed stress-strain curve to lie nearer the stress axis. The greater the time of vulcanisation for any one mixing, the greater, as a general rule, is the temperature rise on stressing. D. F. TWISS.

Joule effect with synthetic rubber. L. HOCK and P. SIEDLER (KAUTSCHUK, 1926, April, 88—90; cf. B., 1925, 816).—Synthetic rubber shows similar elastic properties to natural rubber, which are therefore to be attributed likewise to molecular forces. Stretched unvulcanised "methyl rubber" requires cooling to a lower temperature than natural rubber before it will "freeze," and already recovers its condition at temperatures below 0°; the change, except for the difference in temperature, is analogous in the two cases. Vulcanised synthetic rubber, unlike vulcanised natural rubber which freezes only at very low temperatures, exhibits behaviour similar to that of natural raw rubber and freezes in cold water. Synthetic rubber also resembles natural rubber in that when stretched its condition is fibrous, as can be demonstrated by fracture of stretched samples which have been cooled in liquid air. The results suggest that synthetic rubber is in a lower stage of polymerisation than natural rubber, the freezing behaviour of the latter approaching more closely that of synthetic rubber which has been further polymerised by vulcanisation. The production of synthetic rubber in a higher stage of polymerisation is a problem of importance. D. F. TWISS.

Rubber mixtures with modern reinforcing agents. W. ESCH (Gummi-Ztg., 1926, 40, 1917—1919).—An investigation of the tensile properties of vulcanised rubber mixtures, designed to illustrate the features of "mineral rubber," "thermatomic carbon black," ordinary carbon black, and Dixie

clay as compounding ingredients, using various organic accelerators, *e.g.*, tetramethylthiuram disulphide and diphenylguanidine (cf. Somerville, Indiarubber J., 1925, **69**, 379). D. F. TWISS.

Rubber softeners. W. N. BURBRIDGE (Trans. Inst. Rubber Ind., 1926, **1**, 429—448).—Classification of rubber softeners into two types is suggested, viz., "true softeners" which penetrate the rubber particles, and "pseudo-softeners" which merely lubricate them; the former are commonly cyclic compounds; the latter are commonly aliphatic and more favourable to good ageing behaviour. Results are quoted of experiments with mineral oil, vaseline, naphthalene, rubber resin, rosin, rosin oil, pine tar, pine tar pitch, rape oil, linseed oil, palm oil, olive oil, stearin, stearic, oleic, and palmitic acids, ceresin, and carnauba wax, using a Williams plastometer (B., 1924, 480). The hardness and ease of extension of the vulcanised products are also recorded. The fatty acids show greater softening power than their corresponding glycerides.
D. F. TWISS.

Physical and normal vulcanisation processes, their mutual dependence and their combination. M. KRÖGER (Gummi-Ztg., 1926, **40**, 1803—1807).—The physical process of aggregation (and re-aggregation) is the basis of all processes of vulcanisation; these indeed merely represent various methods for increasing the rate of the aggregation change, *e.g.*, by cold (Le Blanc and Kröger, B., 1925, 932). Vulcanisation by sulphur differs only in minor features, *e.g.*, in the introduction of other components, and in imparting stability over a wider range of temperature. The effect of vulcanisation by hydrogen sulphide and sulphur dioxide is shown to be additive to the physical degree of vulcanisation already existent, increase in the latter necessitating a reduction in the vulcanisation by sulphur for the attainment of comparable tensile properties. The effect of the physical aggregation process (*e.g.*, by storage) on sulphur-vulcanised samples similarly results in displacement of the position of the tensile optimum to a lower proportion of combined sulphur. The observation that the absorption of hydrogen sulphide by rubber is decreased by physical re-aggregation and also by chemical combination with sulphur further justifies the consideration of both processes as forms of vulcanisation. D. F. TWISS.

Factors influencing the weathering of vulcanised rubber. N. A. SHEPARD, S. KRALL, and H. L. MORRIS (Ind. Eng. Chem., 1926, **18**, 615—620).—For comparison of the tendency of strained, vulcanised rubber strips to develop surface cracking when exposed to sunlight, the most advantageous degree of extension is 12½%; greater elongation leads to a larger number of less easily visible fissures. Increase in degree of vulcanisation and the presence of a surface "bloom" of sulphur favour resistance to exposure, and white stocks are more resistant than grey or black. Vulcanised products containing high-grade rubbers, *e.g.*, pale crêpe or smoked sheet, show no advantage over mixtures with "off-colour" rubber. The presence of reclaimed rubber is distinctly favourable. The beneficial influence of the following mineral ingredients (22¼ vols. to 100 vols. of rubber) increases in the order gas black, lithopone, zinc oxide, whiting, barium sulphate, clay, magnesium carbonate. "Mineral rubber," montan wax, rosin, pine pitch, and vegetable oils are of little or no advantage, but paraffin wax is of distinct value, vaseline and mineral oil being successively feebler. The nature of the organic accelerator used in vulcanisation appears to be without influence.
D. F. TWISS.

Swelling of caoutchouc and constitution of the solvent. SALKIND.—See A., June, 576.

PATENTS.

Manufacture of composite material comprising absorbent material and rubber and articles made of or embodying such composite material. E. S. ALI-COHEN (E.P. 250,623, 13.10.24).—Absorbent fibrous material is immersed in latex which has been mixed with a soapy solution, *e.g.*, of an alkali soap or of a mixture of a fatty acid and saponin, at a temperature up to about 70°; the impregnated material is then subjected to the action of a coagulant, *e.g.*, alum, and is washed, pressed, and dried. Powders such as lampblack, zinc oxide, and sulphur, if previously dispersed in the soapy solution, may be introduced into the latex before the impregnation of the fabric. Textile fabrics strengthened by flexible metallic wires may be advantageously treated by this process to form tubes, tyres, or non-extensible belts. D. F. TWISS.

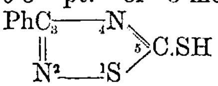
Manufacture of rubber. K. D. P., LTD. (E.P. [A] 250,639, [B] 250,640, 19.12.24. Addns. to E.P. 213,886, B., 1924, 683, 1020).—(A) Concentrated latex paste, produced by the process described earlier, can be mixed with compounding and vulcanising ingredients, these being introduced as powders or suitable suspensions; the proportion of water in the mixture should be kept near 25%. The resulting foamy mass, which still contains the latex in a reversible condition, is then dried. The product can be moulded or extruded at once or after very short mechanical working, and vulcanised when desired. (B) Concentrated latex paste for the preceding process or for re-dilution into latex may be produced by heating latex of rubber, gutta or balata, with a hydrolytic agent, *e.g.*, potassium hydroxide (approximately 2½% calculated on the hydrocarbon content) at 40—80° for one to three hours, and then removing most of the water by evaporation. The hydrolytic agent converts non-hydrocarbon constituents of the latex into protective colloids which enable the water content to be reduced to 15% without coagulation.
D. F. TWISS.

Electrodeposition of rubber. S. E. SHEPPARD and L. W. EBERLIN, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,583,704, 4.5.26. Appl., 8.6.25).—Rubber is deposited on a gas-permeable anode surface from an aqueous suspension, *e.g.*, latex, by passing an

electric current through this surface and through the suspension, the current density being such that gas is evolved adjacent to the surface; the gas is removed from the side further from the cathode.

D. F. TWISS.

Method of vulcanising caoutchouc. L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,583,761-3, 4.5.26. Appl., [A] 9.2.24, [B] 23.12.25, and [C] 26.12.25).—The use of (A) thiodiazoles generally or (B) a 1 : 3 : 4-thiodiazole as accelerators for the vulcanisation of rubber is claimed. (C) Rubber is vulcanised in the presence of a zinc salt of a 1 : 2 : 4-thiodiazole and an activator, such as zinc oxide, litharge, lead acetate, or zinc stearate. For example, a mixture of 50 pts. of rubber, 2·5 pts. of zinc oxide, 3 pts. of sulphur, and 0·5 pt. of 5-mercapto-3-phenyl-1 : 2 : 4-thiodiazole



is heated for 30 min. under a steam pressure of 40 lb. per sq. in.

T. S. WHEELER.

Vulcanisation of rubber at low temperatures. FARBENFABR. VORM. F. BAYER & CO., Assees. of W. ZIESER (G.P., 423,101, 11.9.20).—Rubber mixtures containing a piperidinecarbothionolate ("piperidyldithiocarbamate"), zinc oxide, and less sulphur than is customary, *e.g.*, 2—2½%, can be vulcanised rapidly below 100°; the sulphur passes completely into combination and deterioration by "after-vulcanisation" is impossible.

D. F. TWISS.

XV.—LEATHER; GLUE.

Preparation of sheep skins. G. D. MCLAUGHLIN and E. K. MOORE (J. Amer. Leather Chem. Assoc., 1926, **21**, 274—280).—A large percentage of pickled skins are rendered less valuable through improper treatment in fellmongering. The chief defect is roughened grain surface. Skins which have much fat adhering to them should have this removed before soaking, otherwise they will appear wrinkled. Skins which require transporting to a fellmongery should be washed and brined before they are cured. Dried skins should be soaked in a 15% salt solution for 15—24 hrs., then placed in fresh water for 15 hrs., the water being run away and renewed after 7 hrs. Fresh skins require 5 times their weight of water for proper soaking. After 4 hrs. soaking the grain roughness begins to appear, and 18 hrs. soaking causes serious damage. The best period for salted skins is 10 hrs. at 20° using 6 times their weight of water. The roughened grain condition becomes more marked with rise of temperature above 20°.

D. WOODROFFE.

Bacteriology of goat skin soaking. G. D. MCLAUGHLIN and J. H. HIGHBERGER (J. Amer. Leather Chem. Assoc., 1926, **21**, 280—294; cf. B., 1925, 682).—Dry salt-cured skins are the most heavily contaminated with bacteria, sun-cured skins the least, while green-salted skins are intermediate. The soaks of green-salted skins contain more nitrogen and salt than those of dry-cured. The native "Khari salt" (Indian) consists chiefly of sodium sulphate, and has little or no germicidal effect upon skin. Bacteria on dry salt-cured skins are inhibited least by salt, those from sun-cured skins are inhibited most. Of the bacteria on goatskins 30—80% are proteolytic. The lag periods for skins soaked in 20 pts. of water at 20° were: sun-cured 14 hrs, dry-salted 9 hrs., and green salt-cured 20 hrs. Rise of temperature shortens the lag period and counteracts the inhibitive effect of the salt. The lag period was diminished slightly by decreasing the proportion of water used in soaking green-salted skins. Drumming the skins with change of water before soaking greatly decreased the subsequent bacterial growth. The longer the skins are drummed prior to soaking, the lower will be the concentration of salt in the soak. The presence of magnesium salts in the natural water used for soaking greatly stimulates bacterial growth; iron salts or sodium sulphide reduce it.

D. WOODROFFE.

Utilisation of by-products of saccharin manufacture in the production of synthetic tannins and in tanning. W. HERZOG (Collegium, 1926, 203—208).—*p*-Toluenesulphonyl chloride is used in alkaline pyridine solution to convert tetramethyl-*d*-catechin into an ester, which after treatment with anhydrous hydrazine decomposes into phloroglucinol dimethyl ether and 3-3′ : 4′-dimethoxyphenylpyrazoline. *p*-Toluenesulphonyl chloride combines with tetramethyl-*l*-epicatechin to yield a tetramethylanhydroepicatechin closely related to the benzylcoumarones. *p*-Toluenesulphonyl chloride can be used according to G.P. 297,187–8 (cf. B., 1917, 895) in the production of synthetic tannins, and can be combined with sulphite-cellulose waste liquor in alkaline solution to form ester tannins. The most important by-product is *p*-toluenesulphonic acid which according to G.P. 349,727 forms insoluble products when heated, and these when solubilised have tanning properties. Condensation products of ketones and polyhydric phenols containing no sulphonic groups have excellent tanning properties when dissolved in solutions of the sodium salt of *p*-toluenesulphonic acid.

D. WOODROFFE.

Importance of free fatty acids in fish oils for chamoising. L. KLENOW (Collegium, 1926, 201—203).—A sample of fish oil having iodine value 153 and free fatty acids 4·08% was found to be inferior to another sample having a lower iodine value (135) but higher acid content (16·4%). Addition of lactic acid to the former sample improved its chamoising properties. Tests made at different p_H values showed that chamoising did not take place at p_H 6·0. A sample of delimed pelt at p_H 7·5 tanned well with fish oil having iodine value 143·4 and 8·3% of free acid. Whether pelts for chamoising should be delimed or not, depends on the free acid content of the fish oil used. The experiments show that the presence of free fatty acids in the fish oil facilitates the chamoising process.

D. WOODROFFE.

Does chromium combine with the basic or acidic groups of hide protein? A. W. THOMAS

and M. W. KELLY (J. Amer. Chem. Soc., 1926, 48, 1312—1319).—The rate of combination of chromium with hide substance is greatly reduced if the latter contains either combined quinone or vegetable tannin. Deaminised hide powder also fixes chromium more slowly than the untreated material. These phenomena appear to indicate that the nitrogen groups of the protein play a significant part in chrome tanning, which may thus take place through the formation of ammino-complexes.

F. G. WILLSON.

Hydrolysis of acid sulphate of chrome leather. J. A. WILSON and G. O. LINES (J. Amer. Leather Chem. Assoc., 1926, 21, 299—302).—A sample of freshly tanned, neutralised and washed chrome calf was cut up into small pieces and extracted in two Wilson–Kern extractors. Distilled water was run continuously through the leather in one extractor and tap water through the other. Samples of the leather were withdrawn after periods of 1 to 64 days, analysed for hide substance, total and neutral SO_3. The results showed that practically all the combined acid sulphate in chrome leather is hydrolysable and will be given up as free sulphuric acid either to distilled water or to tap water. The chromium-collagen compound is resistant to washing.

D. WOODROFFE.

Properties of shoe leather. IV. Strength, stretch, and stitch tear. J. A. WILSON and G. DAUB (J. Amer. Leather Chem. Assoc., 1926, 21, 294—299).—Tests have been made on 18 different light and heavy leathers (cf. B., 1926, 504). Patent side was the weakest and kangaroo the strongest. Vegetable calf stretched least and shark most.

D. WOODROFFE.

PATENTS.

Artificial tanning substance. BADISCHE ANILIN- & SODA-FABR., Assees. of O. SCHMIDT (U.S.P. 1,583,801, 11.5.26. Appl., 24.8.25).—Charcoal containing at least 9% of oxygen, calculated on the ash-free material, is oxidised with nitric acid, and the product, after partial neutralisation with a base which forms a soluble salt, is mixed with a natural tanning extract.

L. A. COLES.

Production of water-soluble, sulphonated condensation products [tanning agents] from aldehydes and aromatic hydrocarbons and their derivatives. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of A. VOSS (G.P. 423,033, 4.6.22).—Water-soluble products suitable for use in tanning are obtained by condensing aromatic hydrocarbons or other compounds with sulphonated aromatic aldehydes, in the presence of concentrated acids as catalysts. Suitable products are obtained, *e.g.*, from phenol, cresol, *pp'*-dihydroxydiphenylamine, naphthalene, or phenanthrene on the one hand, and benzaldehyde-*o*- or *m*-sulphonic acid or -2 : 4-disulphonic acid on the other.

L. A. COLES.

Manufacture of glue, gelatin, etc. L. BIERLING, JUN. (E.P. 251,523, 13.10.25).—The raw material is treated in apparatus consisting of a receiver or tank heated by water. Several free spaces in the form of passages are arranged in the tank, and suspended in these spaces are containers filled with the material and provided with metallic discs which may be heated by the water jacket surrounding them. The lower ends of the free spaces are closed by filters and filters are inserted into the lower ends of the containers.

D. WOODROFFE.

Manufacture of glue from chrome leather. ELLENBERGER & SCHRECKER, and O. HUPPERT (G.P. 426,471, 5.5.23).—Chrome leather reduced to shavings or powder is boiled with 0·02—0·1 N-acid, sufficient lime is added to precipitate the chromium and give a solution having an alkalinity of 0·05—0·01 N, and the product, either immediately or after keeping, is boiled with water, yielding glue solution from which insoluble chromium and calcium compounds are removed by filtration.

L. A. COLES.

Condensation products of phenols and acetaldehyde (G.P. 422,904).—See XIII.

XVI.—AGRICULTURE.

Effect of sulphur on the microflora of the soil. J. M. FIFE (Soil Sci., 1926, 21, 245—252).—Addition of sulphur to certain Utah soils under laboratory conditions increased ammonifying power by about 100% where dressings equivalent to 400 lb. per acre were given. Smaller increases were obtained where larger dressings were used. Nitrification was increased by 100% in some cases, but the results were irregular. No effect on azofication was observed. Bacterial numbers increased during the incubation periods. Relative to untreated soils, bacterial numbers were increased by sulphur treatment in the case of a soil rich in organic matter, but were decreased in the case of soils with low organic matter. From 36 to 89% of the added sulphur was oxidised in 30 days, the amount depending on aeration.

G. W. ROBINSON.

Mitscherlich's method for determination of manurial requirement of soils. GERLACH (Z. Pflanz. Düng., 1926, B5, 218—219; cf. B., 1925, 220, 731).—A reply to Mitscherlich (cf. B., 1926, 70). The application of the results of pot experiments to field conditions is criticised.

C. T. GIMINGHAM.

Phosphoric acid requirements of German arable soils. O. LEMMERMANN and H. WIESSMANN (Z. Pflanz. Düng., 1926, B5, 220—230).—The results of a large number of field experiments with superphosphate and basic slag on a variety of crops are tabulated. Of the soils tested, about 25% respond markedly to phosphatic manuring, 13% show a slight response, and 60% show no response.

C. T. GIMINGHAM.

Determination of easily available phosphoric acid in soils by the newer methods. O. ENGELS (Z. Pflanz. Düng., 1926, B5, 208—213).—Lemmer-

mann's method for determining the phosphoric acid requirement of soils by the "relative solubility" (ratio of citric-soluble to total P_2O_5) gives results which are in accord with the response of the soils to phosphatic manuring. Good agreement between the results obtained by this method and by Neubauer's seedling method are shown, taking as the limits below which a soil may be expected to respond to phosphatic manuring, a "relative solubility" of 25 for the former and a figure of 6 mg. P_2O_5 per 100 g. of soil for the latter method.
C. T. Gimingham.

Ion exchange in relation to soil acidity. W. P. Kelley and S. M. Brown (Soil Sci., 1926, **21**, 289—302).—A discussion, mainly theoretical, of the problem of soil acidity. The acidity of mineral soils is held to be primarily due to the presence of acid alumino-silicates. It is suggested that soils could be completely freed of the bases combined with such alumino-silicates by means of electrodialysis. The electronegative colloidal character of soils is due partly, at least, to the ionisation of true chemical compounds rather than to the effect of adsorbed ions. The effects obtained by treating soils with solutions of aluminium chloride are attributable to the hydrogen ions which originate by hydrolysis and not to the aluminium ions themselves. The hydrogen-ion concentration of a soil depends on the dissociation and hydrolysis of its complex alumino-silicates and also on its buffering property.
G. W. Robinson.

Apparatus for the determination of hygroscopicity [of soils etc.]. S. Gericke (Z. angew. Chem., 1926, **39**, 611—612).—For the rapid drying of substances such as soil a phosphorus pentoxide vacuum desiccator is used, the lower part of which consists of a glass hemisphere in order that it may be immersed in a boiling water-bath. Drying is complete in 4 hrs.
A. Geake.

Effects of soil treatments on the draught of ploughs. F. L. Duley and M. M. Jones (Soil Sci., 1926, **21**, 277—288).—In dynamometer experiments on a silt loam it was found that applications of farmyard manure had little effect on the draught of ploughs except a slight tendency to increase it. A more friable seed bed was obtained with the unmanured soils than with those dressed with farmyard manure. Artificial fertilisers have little effect and the draught of ploughs in such cases is slightly less than on soils dressed with farmyard manure. In general the draught increases with decrease in moisture content. As the farmyard manure plots were generally moister than the untreated plots, the differences in draught would have been greater if the tests had been made at the same moisture content.
G. W. Robinson.

[Determination of] potash in mixed fertilisers. G. S. Thorpe (J. Assoc. Off. Agric. Chem., 1926, **9**, 192—193).—A method which renders the phosphoric acid insoluble before the potash is extracted, avoids the precipitation with ammonia and ammonium oxalate, and does not involve ignition, consists in mixing the sample with calcium carbonate and water, keeping for an hour, filtering, washing with hot water, and determining the potash by the platinum chloride method after twice evaporating to dryness with hydrochloric acid and redissolving in hot water.
C. O. Harvey.

"Asahi-Promoloid." E. Blanck and F. Scheffer (Z. Pflanz. Düng., 1926, **B5**, 214—217; cf. Blanck and Alten, B., 1925, 80; Lemmermann and Wiessmann, B., 1925, 562).—No significant increases of yield of dry matter were obtained by the use of "Asahi-Promoloid" in vegetation experiments with radishes, peas, and oats.
C. T. Gimingham.

Principles of the manuring of meadows. H. Raum (Z. Pflanz. Düng., 1926, **B5**, 193—207).—Recent work in Germany on the manuring of meadow land is reviewed and discussed.
C. T. Gimingham.

Influence of nitrate nitrogen on the protein content and yield of wheat. E. Burke (J. Agric. Res., 1925, **31**, 1189—1198).—Plots alternated with fallow and crop contain more nitrate nitrogen during the cropped year, produce more spring and winter wheat per acre, with both straw and grain of a higher nitrogen content, and contain on an average, more moisture to a depth of 3 ft., than plots of the same soil type cropped continuously. Nitrate nitrogen in the alternate crop and fallow plots shows a greater decrease (in parts per million) in the cropped years than in plots cropped continuously. This decrease starts before the wheat plants are big enough to take much plant food from the soil and must be explained largely on the theory of leaching. The continuation of the decrease is probably due to the plants taking nitrate from the soil faster than it was formed.
C. P. Stewart.

Effect of varying the nitrogen supply on the ratios between the tops and roots in flax. T. W. Turner (Soil Sci., 1926, **21**, 303—306).—In the case of barley and maize, increase in the supply of nitrate in culture solutions increases the ratio of stem and leaf to roots. In the case of flax, however, no appreciable increase in the ratio is obtained.
G. W. Robinson.

Form of legume nitrogen assimilated by non-legumes when grown in association. J. H. Stallings (Soil Sci., 1926, **21**, 253—276).—Data are given for dry weight, total nitrogen, ammonia-, nitrite-, nitrate-, and amino-nitrogen of wheat and soya beans grown separately and in association, in sand cultures and in soil. Under favourable conditions wheat can obtain considerable amounts of nitrogen from soya beans grown in association without lowering the nitrogen content of the latter. Wheat plants stimulate ammonia production, assimilation, or accumulation in soya beans. Soya beans fix large amounts of nitrogen when grown in inoculated nitrogen-free sand.
G. W. Robinson.

See also A., June, 647, **Influence of light on absorption by plants of phosphoric acid and potassium** (NEMEC and GRACANIN); **Development of castor-oil plants under the radioactive influence of thorium-*X*** (AVERSENQ, JALOUSTRE, and MAURIN); **Value of litmus, bromocresol-purple and Janus-green milk in a study of nodule organisms of *Leguminosæ*** (STEVENS).

Base exchange and the origin of coal. TAYLOR. —See II.

PATENT.

Soluble oils (U.S.P. 1,582,257).—See II.

XVII.—SUGARS; STARCHES; GUMS.

Inversion and saccharification of beet diffusion juice. N. L. KARAVAEV and A. P. PALKINE (Bull. Univ. Asie Central [Tachkent], 1926, 10, 125—128).—Drying the sugar beet (whether at 100° for 18 hrs. with a residual moisture content of 0·25% or by exposure to the sun for seven days with a residual moisture content of 2·4—2·8%) caused no inversion of the sugar. The sugar can easily be extracted from the dried beet by diffusion; the sugar content of the diffusion juice is higher than in that from raw beet. C. P. STEWART.

See also A., June, 602, **Constitution of maltose** (IRVINE and BLACK; COOPER, HAWORTH, and PEAT); **Identity of *iso*maltose with gentiobiose** (BERLIN).

PATENTS.

Continuous crystallisation of sugar. F. KESSLER (E.P. 245,712, 25.3.25. Conv., 7.1.25).—An apparatus for working sugar syrup designed to maintain uniform supersaturation and to avoid settling out, comprises a series of crystallising tanks arranged in step fashion and fitted with horizontal spiral stirrers, the tanks being interconnected by pipes leading from the bottom of one tank to the bottom of the next. Provision is made for the continuous addition of water or syrup of the correct consistency, so that the proper limits of supersaturation may be maintained in each tank. C. O. HARVEY.

Production of adhesives [vegetable glue]. A. LESZYNSKI (G.P. 424,391, 18.4.23).—Starch or material containing it, is hydrolysed by treatment at about 45° with calcium chloride or magnesium chloride, or mixtures of these, or solutions containing them, the calcium and magnesium are converted into insoluble compounds and removed, and the product is dried, *e.g.*, on hot rollers. L. A. COLES.

XVIII.—FERMENTATION INDUSTRIES.

Modification of malt in relation to the stability of beer. F. E. B. MORITZ and D. H. F. FULLER (J. Inst. Brew., 1926, 32, 269—278).—The ultimate stability of a beer is decided not so much by the amount of nitrogen in the wort which may ultimately become assimilable, as by the amount of nitrogen which is immediately assimilable at the time of pitching. The content of amino-acids in the wort is taken as indicative of the immediately assimilable nitrogen. Worts exactly similar except for varying amounts of amino-acids and pitched with yeast and a measured amount of bacterial suspension, produce beers in which the growth of bacteria is greater and the stability less the greater the amino-acid content of the wort. All malts form amino-nitrogen in a regular manner during modification, but the increase does not continue from start to finish. Some malts form a greater, and some a less, amount of amino-nitrogen under identical flooring conditions. This formation of much or little amino-nitrogen appears to be a characteristic of the barley. In the majority of cases a high percentage of ready-formed sugars in malts will indicate a high percentage of amino-nitrogen, but it does not follow that this will invariably be the case when individual samples are examined. C. RANKEN.

Methods of decarbonating brewing water. W. WINDISCH (Woch. Brau., 1926, 43, 169—170, 181—185).—The precipitation of alkaline-earth carbonates from brewing waters by boiling is costly and not always very successful, and boiling under pressure gives results which are little if any better. Equally good results may be obtained by heating to 80°, at which temperature bicarbonates decompose, but to ensure complete decomposition, the liberated carbon dioxide must be swept out of the water by a current of air. The cheapest and most effective method is by treatment with lime water in the cold, but much greater care is required than in the softening of boiler feed water. Clear lime water of known titre is added in amount just sufficient to combine with free carbonic acid, convert bicarbonates into normal carbonates, and by double decomposition convert magnesium carbonate into hydroxide. After thorough mixing for 5 min. by means of propellers or air the water should be left for about 24 hrs., since the precipitated magnesium hydroxide is at first colloidal. The water is decanted from the precipitate without filtration. Continuous automatic liming processes in which "saturated" lime water is used without titration are not recommended for the treatment of brewing waters. J. H. LANE.

Nitrogen content of worts and their beers. L. G. SMITH (J. Inst. Brew., 1926, 32, 220—225).—The author holds that no nitrogenous substances of maize are rendered soluble when the latter is mashed with barley malt. With varying original gravity, there are slight differences of wort nitrogen content which are in keeping with the known percentages of malt adjunct. The elimination of nitrogen during brewery fermentation is not a question of an actual amount but of a percentage of the total permanently soluble nitrogen of the wort. The actual amounts eliminated differ according to the system of fermentation, type of yeast, rate of pitching, etc., but, apparently, during fermentation, 33% of the permanently soluble nitrogen of the wort is eliminated independently of the actual amount. The actual amount,

however, is a measure of the yeast crop. The amount of permanently soluble nitrogen expressed as a percentage on wort solids is sufficient for a satisfactory fermentation when 100 mg. per 100 c.c. of wort does not exceed 35% of the total. There seems to be a connexion between the rate of elimination of nitrogen and the amount of nitrogen necessary for the proper conduct of a fermentation. Elimination is rapid in the early hours even when the wort content is low. From the nitrogen content of the finished beer, and assuming that the average elimination of nitrogen is 33%, the permanently soluble nitrogen of the wort can be arrived at sufficiently closely to compare with an average all-malt permanently soluble nitrogen figure. Using those figures, indications can be obtained of the amount of nitrogen-free substitutes used in brewing the beer. C. RANKEN.

Buffer substances in wort and beer. P. KOLBACH (Woch. Brau., 1926, **43**, 123—129, 135—140).—The buffer system : primary phosphate–secondary phosphate is most effective between p_H 6 and 7·6, and as a malt mash made with distilled water would have a reaction of about p_H 6 the phosphates in malt are of value in counteracting the alkalinity of a brewing water in the mash tun. In worts and beers almost all of the phosphates present are in the primary form. The system carbonic acid–bicarbonate is important during fermentation and in the beer. The carbon dioxide formed during fermentation may raise the hydrogen-ion concentration of the wort considerably, *e.g.*, from p_H 5·87 to 5·15, and this probably helps to repress bacterial growth during the earlier stages of fermentation before the yeast has produced other acids. In the determination of the p_H value of fermenting liquids the carbon dioxide should not first be removed, as it has an appreciable effect when the reaction is on the alkaline side of p_H 4·5 ; in more acid liquids its effect is relatively unimportant. The organic acids in wort and beer are present in too small amounts for their salts to exercise any very important buffer action. According to Windisch the total buffer action of malt wort cannot be accounted for by the substances already mentioned. Probably an important part is played in this respect by proteins, polypeptides, and amino-acids, but little of a quantitative character is known about the buffer action of these substances.
J. H. LANE.

Oxygen as a factor in the production of sound beer. H. HERON (J. Inst. Brew., 1926, 32, 261—269).—Aeration of worts exerts a very considerable influence on brewery fermentations and on the health and longevity of the yeast. The principal factors which have a distinct bearing on the degree of aeration are the type and height of refrigerators, the rate at which the wort is run over them, and the presence of steam in the refrigerator room. Deficiencies in the aeration of worts below 85% of the maximum have an appreciable effect upon the health, vigour, and keeping properties of yeast, with the exception, perhaps, of fermentations carried out on the Yorkshire Stone Square system. Although the results of original oxygen deficiency in worts cannot be rectified to any appreciable extent by subsequent rousing during fermentation, regularity in attenuation and more healthy yeasts are produced by aerating the worts with pure air while they are running into the fermentation vessels. Small traces of oxygen influence the conditioning of beer after racking. In the case of cask beers and naturally matured bottled beers, its influence is favourable and highly necessary, but it is very undesirable in the case of chilled and filtered beers. C. RANKEN.

Amylase type : neutral salts—amino-acids—peptone. H. HAEHN and H. BERENTZEN (Woch. Brau., 1926, **43**, 91—93, 101—104).—A neutral solution containing sodium, potassium, and calcium chlorides, alanine, leucine, and Witte's peptone, mixed with starch solution and incubated at 40°, under sterile conditions, exerted a feeble hydrolytic action, the iodine reaction of the solution changing from blue to yellow in less than 24 hrs., although the production of sugar was too small to be demonstrated. A still feebler action was exerted by the inorganic salts alone (cf. Biedermann, B., 1923, 620 A). In an experiment continued for 31 days, with successive additions of starch solution, it was calculated that 12 g. of starch yielded 1·34 g. of maltose, 2·42 g. of fermentable dextrin, and 8·28 g. of unfermentable non-reducing dextrin containing some starch. The hydrolysis appears to be a true catalysis, but it does not take place in complete absence of oxygen. (See also Petit, B., 1925, 776.) J. H. LANE.

Assimilation, respiration, and fermentation. W. WINDISCH (Woch. Brau., 1926, **43**, 159—162, 170—173, 188—191).—A review of recent work.
J. H. LANE.

Citric acid content of Grecian musts. S. GALANOS (Z. Unters. Lebensm., 1926, **51**, 217—220). —The Denigès reaction as modified by Muttelet (B., 1923, 1240 A) is sufficiently sensitive for the detection and determination of citric acid in wines and musts. Of 11 samples of Grecian musts, two contained only traces of citric acid, whilst the others contained quantities ranging from 0·1 to 0·4 g. per litre. B. W. CLARKE.

Determination of lactic acid in wine. G. BONIFAZI (Mitt. Lebensmittelunters. Hyg., 1926, **17**, 9—14 ; Chem. Zentr., 1926, I., 3365).—Wine (25 c.c.) is neutralised with saturated barium hydroxide solution, 2·5 c.c. of 10% barium chloride solution are added, and the liquid is diluted to 100 c.c. with 95% alcohol, shaken, and allowed to settle. After filtration, 80 c.c. are evaporated to dryness, and the residue is calcined and dissolved in 20 c.c. of 0·1 *N*-hydrochloric acid. The quantity of lactic acid plus acetic acid is calculated from the volume of acid neutralised, and the quantity of acetic acid is determined by the usual method, the difference giving the lactic acid. Sulphur dioxide, if present, must first be removed. L. A. COLES.

Character in pot still whisky. S. H. Hastie (J. Inst. Brew., 1926, 32, 209—220).—Among the many factors determining "character," which is defined as the palate flavour produced by pot still whisky, the use of peaty waters for mashing, malt subjected to a process of peat smoking in the earlier stages of drying with subsequent curing over a peat fire, and the time during which the wash after fermentation remains in the wash charger commanding the first still play an important part. It is further influenced by the maintenance or otherwise of the temperature of the wash subsequent to transference from the wash back, by the degree of fermentation attained by the wash prior to removal, and by the strength and virility of the ever-present bacterial contamination of vessels and materials due to the use of unboiled wort. Considerable character formation occurs during the primary distillation owing to the interaction of the alcohols, acids, and esters in the wash, and, in addition, the effect of heating the yeast residue etc. over a direct fire is of considerable moment. Furfuraldehyde, an important character-producing substance, is absent from the wash prior to distillation. It is produced in the wash still as the result of the action of acids on pentoses under the influence of heat, the system of heating the still being a governing factor. Character is further modified in the spirit still by the interacting substances and by the degree of separation obtained during fractionation. There is a slight and varying gain of desirable character in whisky during maturation. C. Ranken.

See also A., June, 640, **Definition of enzymic activity of enzymic preparations** (Von Euler and Josephson); **Enzymes of emulsin** (Josephson). 641, **Inulase** (Pringsheim and Perewosky); **Hydrolysis of sucrose solutions by invertase** (Ingersoll); **Cultivation of yeast in a synthetic medium** (Aubel, Genevois, and Salabartan); **Assimilation of nitrogen by yeast from culture media in the aeration process** (Claassen); **Action of manganese on alcoholic fermentation** (Rosenblatt and March). 642, **Influence of fatty acids and their salts on alcoholic fermentation by living yeast** (Katagiri); **Carbohydrate and fat metabolism of yeast** (Maclean and Hoffert); **Action of yeast on lactic acid** (Hoffert); **Mechanism of anti-ketogenic action of yeast** (Weiss and Altai); **Action of previous treatment of yeast on affinity constant of invertase** (Von Euler and Josephson). 643, **Toxic effects of potassium chloride on lactic acid bacillus** (Bachrach).

Patents.

Dehydration of impure ethyl alcohol. Distilleries des Deux-Sevrés (E.P. 243,368, 19.11.25. Conv., 20.11.24).—If the process for the dehydration of purified alcohol described in E.P. 214,581 (B., 1925, 185) is applied to the dehydration of impure commercial alcohols containing impurities more volatile than alcohol, such impurities collect at the top of the distilling apparatus and cause the decanting device to stop. The present process is based on the use of azeotropic mixtures. Part of the decanted layer containing the major part of the impurities is continuously removed and distilled in an auxiliary distilling apparatus, the purified liquid being returned to the ordinary apparatus at a suitable point. The condensed impurities are washed with water if necessary and the mixture of water and impurities is separated by distillation in a second auxiliary apparatus. The process may be modified by the addition of water to the layer containing impurities in the decanting apparatus in order to effect decantation of the entraining body which is returned to the main distilling apparatus. C. Ranken.

Process for separating enzymes from their solutions. S. Sokal. From Kalle u. Co. A.-G. (E.P. 251,405, 24.3.25).—The enzymes are separated by first saturating their solutions more or less completely with a neutral salt, *e.g.*, common salt, and then precipitating the enzymes by means of a water-soluble salt of an aromatic sulphonic acid, especially a salt of naphthalene-1-sulphonic acid. The precipitate contains the enzyme in combination with the sulphonate. The process may be modified by adding sodium phosphate together with calcium chloride and so producing a precipitate in the solution prior to the addition of the sulphonate. The enzyme, precipitated in combination with the calcium phosphate and sulphonate salt, is dissolved in water and freed from the calcium phosphate. Finally, the enzyme is reprecipitated together with the sulphonate by saturating the filtrate with common salt.
C. Ranken.

Yeast manufacture. J. F. Wroten, Assr. to Liberty Yeast Corp. (U.S.P. 1,580,550, 13.4.26. Appl., 20.8.24).—Soured, filtered, and sterilised wort is diluted to 1·5° Balling and seed yeast added, the yeast being fed with portions of the more concentrated wort and additions of "bone precipitate" (acid calcium phosphate) as required.
E. S. Kreis.

Manufacture of alcohols and acetone [by fermentation]. E. H. Strange (E.P. 251,678, 10.1.25).—See U.S.P. 1,550,928; B., 1925, 897. The mash may be made from molasses or Jerusalem artichokes.

XIX.—FOODS.

Refrigeration of meat. I. Beef. G. A. Cook, E. F. S. Love, J. R. Vickery, and W. J. Young (Australian J. Exp. Biol. Med., 1926, 3, 15—31). Increasing the latent period of freezing increased the changes in the beef as determined by the quantity of drip, the nitrogen loss on thawing, and the microscopical appearance of the frozen meat. Microscopical examination showed that with a longer latent period of freezing, ice-crystals were fewer but larger, there were more bands of compressed "dehydrated" fibres and fewer undistorted fibres than in rapidly frozen meat. Further, on thawing, autolysis proceeded more rapidly and to a greater extent. Usually

younger muscle could be frozen and thawed with less change than that from older beasts. The quantity of drip was decreased by slow thawing and also by storage at 1° after thawing, showing that imbibition of liquid by the colloid continued after thawing was complete. In general, too, the drip was less if the meat was stored at 1° for not more than ten days before freezing. Beef showed much more ice formation and distortion than mutton frozen under similar conditions. Of the conditions tested—rate of freezing, rate of thawing, time of storage before freezing and after thawing, and temperature during the freezing process—the first appears to be the most important, though none is without effect.

C. P. Stewart.

[Flour] ash; determination and significance. C. O. Swanson (Amer. Miller, 1926, **54**, 70—71; Chem. Abstr., 1926, **20**, 1284).—As wheat ripens, the proportion of bran to endosperm, and hence the total percentage of ash, decreases. Light test weight wheat contains more ash than heavy. A flour with a high percentage of ash is darker in colour than a low-ash flour, owing to the presence of finely divided bran, pulverisation of which may be prevented by suitable humidity control. Wheat is deficient in calcium.

A. A. Eldridge.

[Flour] ash. H. L. Thompson (Amer. Miller, 1926, **54**, 160; Chem. Abstr., 1926, **20**, 1284).—A typical analysis of ash of a patent flour is: P_2O_5 50, K_2O 35, CaO 7, MgO 7, Na_2O 0·38, Fe_2O_3 0·5%. In the lower grades the magnesium is higher and the calcium lower. There is no close correlation between ash content and baking quality, but the determination of ash is of value in the control of the operation of a mill.

A. A. Eldridge.

Reductase of milk. C. Barthel (Arkiv Kemi, Min., Geol., 1926, **9**, No. 19).—The decolorisation of methylene-blue in the presence of sterile milk takes place equally rapidly with either succinate or citrate as hydrogen donator. No reduction takes place if a mixture of lactose, kaolin, and peptone replaces the milk; but if to this system, salts are added in the proportion in which they are present in milk, then hydrogen transport and decolorisation occur (cf. B., 1926, 105).

C. Rimington.

Analysis of butter. L. C. Mitchell and S. Alfend (J. Assoc. Off. Agric. Chem., 1926, **9**, 209—220).—Official methods for the determination of fat are criticised and the sources of error reviewed. The Gooch method is condemned on the ground that some fat is retained by the asbestos and a similar method employing white sand in place of asbestos is proposed, the extraction being carried out with carbon tetrachloride (in place of light petroleum) in a special closed-system extraction apparatus. Results are in agreement with those obtained by the official method (cf. B., 1925, 823).

C. O. Harvey.

Physiology of apples. V. Methods of ash analysis, and effect of environment on the mineral constitution of the apple. J. W. Brown (Ann. Bot., 1926, **40**, 129—147).—Methods suitable for the determination of total ash, potassium, calcium, magnesium, iron, and phosphate in apples are described. The amounts of the mineral constituents present differed significantly in apples of the same variety grown on different soils and on different stocks on the same soil. Good keeping qualities were associated with high percentages of potassium and phosphate. Apples affected with "bitter pit" showed high ash values and a low percentage of phosphate in the ash.

C. T. Gimingham.

Relative loss of weight of white Calville apples during storage. G. Rivière and G. Pichard (Bull. Soc. chim., 1926, [iv.], **39**, 802—803).—A white Calville apple weighing 142·45 g. when kept under a bell for 177 days lost 4·91 g., of which 4·26 g. was water lost by evaporation and the remainder water and carbon dioxide from oxidation of carbohydrates.

W. Thomas.

Microscopical determination of husk in cacao products. C. Griebel and F. Sonntag (Z. Unters. Lebensm., 1926, **51**, 185—198).—The finely ground material is first bleached by means of sodium hypochlorite, and then stained by carbofuchsin to increase the contrast between the husks and the cocoa substance proper; it is then evaporated to dryness with potassium bromide and a suspension in water is placed in a special type of microscope counting chamber. The number of scleridæ is referred to the weight of fat-free cocoa substance; the maximum should be 390 per mg., corresponding to 3% of husk in the fat-free cocoa or 1% in the beans. Venezuelan cocoas are characterised by a low proportion of husks.

B. W. Clarke.

Natural and caffeine-free coffee. J. Pritzker and R. Jungkunz (Z. Unters. Lebensm., 1926, **51**, 97—114).—The water-soluble matter in coffee, upon which the properties of the beverage depend, is increased by strong roasting, while the acidity and moisture content are decreased. A method and apparatus for determining the moisture content by distillation with xylene are described and it is suggested that the present standard of 3% maximum moisture content should be raised to 8%, since coffee with an original moisture content less than 3% often reaches 7% of moisture on keeping, even when put up in packets. A modification of the method of Fendler and Stüber (B., 1914, 802) for the determination of caffeine is described, the extraction with ammonia and chloroform being performed by boiling for 1 hr. under a reflux condenser. The caffeine content of various brands of coffee labelled caffeine-free or poor in caffeine was found to range from 0·72 to 2·43%. Caffeine-free coffee is prepared by treating the coffee bean with steam and then extracting with benzene or other solvents and finally washing, drying, and roasting. The nature and extent of the preliminary and final treatments affect the percentage of water-soluble matter, which in some brands of caffeine-free coffee is 16% instead of 20—30%, which is the

standard for ordinary coffee; it is suggested that a similar standard should be set up for caffeine-free coffee; the maximum allowable percentage of caffeine should also be stated. B. W. CLARKE.

Determination of formic acid in food products. J. K. MORTON and G. C. SPENCER (J. Assoc. Off. Agric. Chem., 1926, **9**, 221—224).—It is proposed to substitute for the steam distillation method of Fincke a process involving distillation with xylene, followed by direct determination of the formic acid gravimetrically with mercuric chloride. Results obtained compare favourably with those obtained by the steam distillation method, and the manipulation is simpler and less tedious.
C. O. HARVEY.

Toxicity of white beans. O. LÜNING and W. BARTELS (Z. Unters. Lebensm., 1926, **51**, 220—228).—Ground white beans contain a poisonous constituent, phasin, which is destroyed or converted into a non-toxic substance on heating. Experiments on mice are described showing that beans cooked by the usual methods are non-poisonous.
B. W. CLARKE.

Determination of allyl mustard oil [allyl thiocarbimide] in mustard flour. L. COLOMBIER (Ann. Falsif., 1926, **19**, 160—169).—The French Codex method for the determination of allyl thiocarbimide in mustard flour in a slightly modified form (cf. Luce and Doucet, B., 1922, 515 A) gave satisfactory results. A distillate of 90 instead of 65 c.c. should be collected, and after precipitating the sulphur of the thiosinamine as silver sulphide, the excess of silver should be titrated with 0·1 *N*-thiocyanate. Unless maceration of the sample (5 g.) is limited to 1 hr. loss of mustard oil must be prevented by addition of 25 c.c. of alcohol, or of sodium fluoride. A modification of Jörgensen's method for the seeds of crucifers (cf. Morvillez and Meesemaecker, B., 1924, 922), whereby the essential oil is distilled into ammonia and the thiosinamine formed weighed gave practically identical results to the modified Codex method. D. G. HEWER.

Water content of tinned sausages. A. GRONOVER and E. WOHNLICH (Z. Unters. Lebensm., 1926, **51**, 137—145).—Meat sausages, sterilised and tinned in brine, take up water slowly, until after about 3 weeks equilibrium is attained. The Feder ratio number (ratio of water to organic non-fatty material) shows that the added or foreign water is increased by up to 20%. The amount of water thus taken up can be decreased by using a stronger salt solution, but this is impossible in practice, since the sausages would be too salt for consumption. This is because the process is one of osmosis which proceeds through the skin of the sausage until an equilibrium is set up both for the salt and the water in the sausage itself and in the surrounding brine. This diffusion and osmosis was studied in various brands of smoked, sterilised, and tinned sausages by the depression of the freezing point in the fluid expressed from the sausage material itself. The results show that sausages preserved in brine must necessarily contain more water than ordinary smoked sausages cooked immediately after the smoking process.
B. W. CLARKE.

Determination of tin and lead in preserves and containers. A. W. OWE (Z. Unters. Lebensm., 1926, **51**, 214—217).—After destruction of organic matter by sulphuric acid and conversion of the tin into metastannic acid, the tin is reduced to the metallic state by the addition of aluminium and hydrochloric acid in an atmosphere of carbon dioxide. The tin is then determined volumetrically by means of iodine and thiosulphate, the results being accurate to within 2·5 mg. Sn per kg. of the preserved product. Lead is determined after destruction of organic matter by sulphuric acid by dissolving the lead sulphate formed in sodium acetate, reprecipitating as lead chromate, and determining by titration with thiosulphate the iodine liberated from an acid solution of potassium iodide. The results are accurate to within 1·25 mg. Pb per kg. of preserve, and the method cannot therefore be applied where the lead content is less than 10 mg. per kg. For the determination of tin and lead in tin-plate the tin is dissolved from a known area of plate by means of sodium peroxide or electrolytically, and the metals are determined by the methods described above, suitably modified.
B. W. CLARKE.

See also A., June, 631, **Binding of acid and alkali by proteins** (GORTNER and HOFFMAN); **Effect of p_H and of temperature on hydrolysis of caseinogen** (CARPENTER); **Membrane hydrolysis of neutral sodium caseinogenate** (STARLINGER); **Alteration of albumins by heat** (SPIEGEL-ADOLF). 633, **Determination of tyrosine and histidine in proteins and of tyramine in mixtures containing protein. Histidine and tyrosine content of various proteins** (HANKE). 643, **Use of low temperatures in preparation of pure proteins** (PARSONS). 644, **Concentrated vitamin-B from brewers' yeast** (SEIDELL); **Antineuritic vitamin** (JANSEN and DONATH); **Production by irradiation with ultra-violet light of antirachitic properties in sterols from small Siak illipé nut** (HUME and SMITH). 645, **Catalytic formation of an antirachitic cholesterol derivative** (BILLS); **Hydrogenated vegetable oil as source of vitamin-E** (KENNEDY and PALMER).

PATENTS.

Manufacture of poultry and animal foods from waste and condemned foods and other food refuse. J. LEWIS (E.P. 250,675, 16.1.25).—The disintegrated raw material is washed with jets of boiling water while passing over a screen, then treated with wet steam in a centrifuge, and dried, say, at 540° by means of superheated steam and air.
A. RAYNER.

Sterilisation and clarification of liquids. W. McCOMB and W. A. HEYMAN (U.S.P. 1,573,177, 16.2.26. Appl., 6.4.22).—To sterilise liquids such as orange juice without agitation and consequent

oxidation, the liquid is passed through a coil heated to 63°, under a pressure of at least 200 lb. per sq. in., and then into a coil heated to not more than 21°, under a pressure of less than 25 lb., and finally into a coil cooled below 1° at atmospheric pressure.

E. S. KREIS.

Manufacture of leavened bread. C. HOFFMAN, Assr. to the FLEISCHMANN Co. (U.S.P. 1,579,447, 6.4.26. Appl., 15.9.23).—Yeast is carefully dried so that the cell walls shall not be broken (which would liberate the amino-acids and make the bread bitter); 5% of this yeast is then sifted into the dough, together with salts to make up for the salts removed when the wheat was screened, to give a bread with a high vitamin-*B* and protein content.

E. S. KREIS.

Flour-treating apparatus. R. H. GUERRANT, Assr. to INDUSTRIAL APPLIANCE Co. (U.S.P. 1,584,924, 18.5.26. Appl., 19.9.23).—An apparatus for treating flour with a gas comprises a vertical cylindrical chamber, tapered at the lower end and provided with a feeding hopper and gas inlet at the upper end. A rotating member surrounded by a cylindrical baffle plate is fixed just below the hopper to assist in the even distribution of the flour throughout the chamber.

A. R. POWELL.

Extracting oil from milk. Obtaining oil and casein from cream. Producing milk oil from sour cream. Emulsifying and converting fats into cream. C. E. NORTH, Assr. to THE MILK OIL CORP. (U.S.P. 1,584,123–6, 11.5.26. Appl., [A] 2.3.23, [B] 19.2.24, [C] 5.4.24, [D] 27.1.25).—(A) Cream is separated from milk, washed, diluted with water, and the suspension heated to coagulate the remaining casein and allow the oil to rise to the surface. The hot oil is filtered and dried. (B) Whipped cream is heated sufficiently to melt the fat and coagulate the curd, the oil and curd being separated by keeping after the addition of water. (C) Sour cream is heated to remove water and cause the fat globules to coalesce, and the oil is freed from curd by filtration. (D) Oil and an emulsifying agent are continuously fed into a mixing vessel, the resulting emulsion being displaced from the vessel by the pressure of incoming materials.

C. O. HARVEY.

Process for bleaching flour. WALLACE & TIERNAN Co., INC., Assees. of J. C. BAKER (E.P. 232,607, 15.4.25. Conv., 21.4.24. Addn. to 159,166).—See U.S.P. 1,510,132; B., 1924, 962.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Keeping properties of aqueous digitalis extract. W. HINTZELMANN and G. JOACHIMOGLU (Arch. exp. Path. Pharm., 1926, **112**, 56—59).—Aqueous digitalis extract deteriorates more rapidly in alkaline than in neutral or acid solution. In all cases acid substances appear to be produced. It is recommended both for analytical purposes and for medicinal use that no addition of alkali or acid be made to the aqueous extract, and that it be standardised soon after preparation.

C. P. STEWART.

[Saccharin chemistry.] W. HERZOG (Z. angew. Chem., 1926, 39, 728—729).—The separation of *o*- and *p*-toluenesulphonamides may be effected by heating with the calculated amounts of lime and water. The mother liquor contains mainly the calcium salt of the *o*-isomeride; it is fractionally precipitated in the cold with concentrated hydrochloric acid and the product purified by alcohol. When 2:4-toluenedisulphonamide is present (its production may be controlled by regulating the initial chlorosulphonation of the toluene) it is precipitated almost pure in the last fraction. The residue from the lime treatment is dissolved in hot water and the *p*-isomeride obtained by similar fractional precipitation. Acidification of the mother liquors obtained by the precipitation of saccharin solution with alkali yields the "bitter oxidation principle," which is obtained in a pure state, m.p. 246—247° (corr.), on repeated crystallisation from water. The sodium salt was also prepared.

S. K. TWEEDY.

See also A., June, 591, **Evaluation of *Ferrum carb. sacchar.*** (VON BRUCHHAUSEN). 597, **Synthesis of dihydrochaulmoogric and dihydrohydnocarpic acids** (NOLLER and ADAMS); **Synthesis of homologues of dihydrochaulmoogric and dihydrohydnocarpic acids** (HIERS and ADAMS). 598, **Constitution of aldehyde and ketone hydrogen sulphite compounds** (RASCHIG). 602, **Preparation of diamines and amino-alcohols** (PUTOCHIN). 604, **Bromination and iodination of organic compounds** (ZMACZYNSKI). 606, ***p*-Phenoxycarbamides and thiocarbamides derived from *p*-phenoxyaniline. Effect of phenoxy-group on taste** (LANGE and REED). 614, **Constitution of civetone** (RUZICKA). 618, **Crystalline Kombe strophanthin** (JACOBS and HOFFMANN). 619, **Hydration of nopinene** (AUSTERWEIL). 627, **Rotation of quinine and its salts** (SCHOORL); **Oxyacanthine** (GADAMER and VON BRUCHHAUSEN). 632, **Determination of ethyl alcohol and its homologues and their separation from other substances** (FISCHER and SCHMIDT). 633, **Identification and determination of cholesterol** (STEINLE and KAHLENBERG). 643, **Wyss chemical method for assay of insulin** (BISCHOFF, MAXWELL, and BLATHERWICK). 644, **Isolation of thyroxine from thyroid gland** (HARINGTON). 646, **Content of various forms of carbohydrates in tobacco** (BALABOOCA).

Oil of *Johannesia Princeps.* ETZEL and KING.—See XII.

Utilising by-products of saccharin manufacture. HERZOG.—See XV.

Determination of allyl mustard oil in mustard flour. COLOMBIER.—See XIX.

Determination of strychnine. Elmore.—See XXIII.

PATENTS.

Production of bornyl esters from pinenes or mixtures containing pinenes for the manufacture of camphor. V. Isajev (E.P. 251,147, 23.7.25).—Material containing pinenes, *e.g.*, turpentine oil, is esterified in the presence of a solvent consisting of a neutral or acid ester of the acid used in the esterification, catalysts being added, if necessary. For example, methyl or ethyl benzoate, methyl, ethyl, or propyl salicylate, or methyl or ethyl phthalate, is used in the production of bornyl benzoate, salicylate, or phthalate, respectively. L. A. Coles.

Preparation of monocyclic ketones with more than nine ring members. M. Naef & Co. (E.P. 251,188, 18.5.25. Addn. to 235,540). The process described in the chief patent (cf. B., 1925, 738) is modified by the use of mixtures of thorium and cerium salts of the carboxylic acids, or by the addition of cerium or thorium, or of thorium oxide, to the acids used for the production of the ketones. Mixtures of different ketones are obtained by heating mixtures of the corresponding dicarboxylic acids (cf. Chuit, A., 1926, 599). The preparation of *cyclo*pentadecanone, *cyclo*decanone, *cyclo*hexadecanone, and *cyclo*octadecanone from tetradecane-$\alpha\zeta$-dicarboxylic acid or its anhydride, thorium nonane-$\alpha\iota$-dicarboxylate, thorium pentadecane-αo-dicarboxylate, and thorium heptadecane-$\alpha\rho$-dicarboxylate, respectively, is described. L. A. Coles.

Manufacture of quinine salts. A.-G. Chem. Werte (E.P. 251,526, 15.10.25. Conv., 8.7.25).—Solutions containing quinine sulphate together with sulphates of other alkaloids, obtained, *e.g.*, by extracting cinchona bark with organic solvents, and treating the residue with sulphuric acid, are heated under 1·5—3 atm. pressure with a sufficient quantity of sodium hydroxide or other alkali to precipitate the alkaloids other than quinine; after removing these, quinine of 98—100% purity is recovered from the mother liquor. L. A. Coles.

Production of iodised pyridine derivatives. A. Binz and C. Räth (E.P. 251,578, 30.10.24).—Compounds with bactericidal properties are prepared by the action of an alkali iodide upon diazotised aminopyridines and their derivatives. For example, 2-chloro-5-iodopyridine, m.p. 99°, and 5-iodo-2-hydroxypyridine are obtained by adding to potassium iodide solution, diazotised 2-chloro-5-aminopyridine and diazotised 5-amino-2-hydroxypyridine, respectively, these being obtained by the reduction of the corresponding nitro-compounds. L. A. Coles.

Choline compound having laxative properties. J. Callsen, Assr. to Winthrop Chem. Co. (U.S.P. 1,580,012, 6.4.26. Appl., 11.5.25).—The hydroxy-group of the intermediate compounds mentioned in U.S.P. 1,518,689 (B., 1925, 1012), which are obtained by treating aliphatic alkylamines with alkylene glycols, is acylated and the product is treated with an alkylating agent, or alternatively the products mentioned (*loc cit.*). hydroxyalkoxyalkyltrialkylammonium halides, are treated with acylating agents to give choline compounds in which the hydroxyl hydrogen of the hydroxyethyl group in choline is replaced by an acylated hydroxyalkyl radical. These compounds are soluble in water and are of value as laxatives for subcutaneous use, having similar properties to arecoline. They are superior in this respect to the compounds described in the prior patent. For example, dimethyl-β-(β-hydroxyethoxy)ethylamine is converted by acetic anhydride into *dimethyl-β-(β-acetoxyethoxy)ethylamine* which has b.p. 103—108°/27mm. and yields with methyl bromide in benzene *trimethyl-β-(β-acetoxyethoxy)ethylammonium bromide*, m.p. 126—128°. This compound is also obtained by treating trimethyl-β-(β-hydroxyethoxy)-ethylammonium bromide with acetyl chloride. The corresponding *iodide* has m.p. 124°. T. S. Wheeler.

Production of esters. D. A. Legg and C. Bogin, Assrs. to Commercial Solvents Corp. (U.S.P. 1,580,143, 13.4.26. Appl., 16.6.23; cf. U.S.P. 1,401,117, B., 1922, 89 A; E.P. 166,249, B., 1921, 614 A).—When n-butyl alcohol is passed at 280—300° over cupric oxide a mixture of butyl butyrate, butyl alcohol, butaldehyde, and water is obtained which is readily separated into its components by distillation. T. S. Wheeler.

Manufacture of butyric acid. D. A. Legg and C. W. Hancock, Assrs. to Commercial Solvents Corp. (U.S.P. 1,580,144, 13.4.26. Appl., 31.7.23).—Crude butyric acid obtained by the oxidation of butaldehyde and containing about 90% of butyric acid and impurities difficult to separate by distillation, is neutralised with a strong solution of caustic soda and the solution filtered and treated with an excess of sulphuric or other mineral acid of such concentration that a saturated solution of the alkali salt of the acid is formed which salts out the butyric acid liberated. The mixture is distilled and the fraction of b.p. 155—165° is separated and distilled in presence of 1% of concentrated sulphuric acid. The distillate is boiled under reflux for 3 hrs. to remove sulphur dioxide and redistilled, when butyric acid of over 98% purity is obtained. Butyric acid obtained by the hydrolysis of butyl butyrate (cf. preceding abstract) with alcoholic caustic soda is purified by distilling off the alcohol, adding water, and treating the sodium butyrate solution as above. T. S. Wheeler.

Production of amide acid sulphates from nitriles. R. B. Trusler, Assr. to Roessler & Hasslacher Chem. Co. (U.S.P. 1,581,621, 20.4.26. Appl., 22.7.25).—Nitriles (including hydrogen cyanide) are hydrolysed by sulphuric acid monohydrate to the sulphate of the corresponding amide, $R\cdot CO\cdot NH_2,H_2SO_4$, in nearly quantitative yield. The reaction is catalysed by the addition of an organic or inorganic halide, and may, if desired, be performed in presence of an inert liquid such as

carbon tetrachloride, and at temperatures from below 0° to above 70°. Whilst monohydroxynitriles such as acetaldehyde cyanohydrin are readily hydrolysed in this way, polyhydroxynitriles are decomposed. Nitriles containing an amino-group are also unsuitable. T. S. Wheeler.

Production of esters from amide acid sulphates. R. B. Trusler, Assr. to Roessler & Hasslacher Chem. Co. (U.S.P. 1,584,907, 18.5.26. Appl., 22.7.25).—Alcohols and amide acid sulphates (cf. U.S.P. 1,581,621, *supra*) react spontaneously or on gentle warming to give the corresponding esters and ammonium hydrogen sulphate in almost quantitative yield. For example, 1 mol. of formamide acid sulphate and 1 mol. of ethyl alcohol are mixed with cooling and subsequently heated to 50° when ethyl formate is obtained in almost theoretical yield, according to the reaction $H.CO.NH_2,H_2SO_4+EtOH=H.COOEt+NH_4HSO_4$. T. S. Wheeler.

Oxidation of ethyl alcohol to acetaldehyde. E. Krause, Assr. to Roessler & Hasslacher Chem. Co. (U.S.P. 1,581,641, 20.4.26. Appl., 4.6.24). —Ethyl alcohol vapour mixed with oxygen, preferably diluted with an indifferent gas, is passed over silver gauze at such a rate that the heat evolved in the reaction maintains the catalyst just incandescent. For example, 20 g. of 96% ethyl alcohol and 25 litres of air per hr. are passed through a quartz tube 18 mm. in diameter, packed for a length of 60 mm. with silver wire discs. Initially the catalyst is heated, but the reaction then proceeds exothermally and a 92% yield of acetaldehyde is obtained. T. S. Wheeler.

Detoxifying compound. G. P. Metz, Assr. to H. A. Metz Laboratories, Inc. (U.S.P. 1,583,002, 4.5.26. Appl., 6.11.25).—Crystalline sodium thiosulphate is heated to 120° with 1, 2, 3, or 4 mols. of carbamide to yield stable crystalline addition compounds, which form stable sterile solutions in water and are of therapeutic value in the treatment of metallic toxemias since they possess the detoxifying action of sodium thiosulphate with the eliminating action of carbamide. The solutions may be injected intravenously without irritation of the tissues. T. S. Wheeler.

Process of obtaining alcohols etc. W. H. Rodebush, Assr. to U.S. Industrial Alcohol Co. (U.S.P. 1,583,314, 4.5.26. Appl., 14.4.21).—The aqueous alcohol is mixed with an ester and the mixture distilled, when a ternary constant-boiling mixture is obtained, the excess alcohol being driven off and recovered almost pure. The constant-boiling mixture is mixed with water when it separates into two layers, the one containing a high percentage of ester and the other containing a low percentage of ester. The first is distilled, yielding a constant-boiling mixture which is added to that from the first distillation, and excess of pure ester for addition to the feed alcohol, and the latter is rectified before addition to the still, where pure alcohol is being prepared. The process is specially described with reference to ethyl alcohol and ethyl acetate, but is not limited with respect to any of the constituents. E. S. Kreis.

Purification of [*iso*]propyl alcohol. H. F. Willkie, Assr. to U.S. Industrial Alcohol Co. (U.S.P. 1,583,328, 4.5.26. Appl., 4.11.21).—Commercial *iso*propyl alcohol is mixed with about half its volume of 2% permanganate solution, or other suitable non-volatile oxidising agent, and distilled, using a reflux condenser down which a 0·04% solution of permanganate is trickling. The first and last 10% of distillate are collected separately and treated with an equal volume of 10% sulphuric acid and re-distilled under the same conditions. E. S. Kreis.

Barbituric acid derivative. W. Ursum, L. Schütz, and L. Taub, Assrs. to Winthrop Chem. Co. (U.S.P. 1,584,156, 11.5.26. Appl., 17.12.24).—An alkyl-, aryl-, or aralkyl-monosubstituted derivative of malonic or acetic acid is converted into the corresponding disubstituted monochlorocrotonyl derivative by condensation with monochlorocrotonyl bromide, and the product is converted into the corresponding barbituric acid derivative by condensation with carbamide. The products possess hypnotic properties when administered in doses of 0·25—0·5 g., have no narcotic action and no deleterious secondary action. The alkali salts give stable non-irritant aqueous solutions. An alternative method of preparation consists in forming a monosubstituted monochlorocrotonyl derivative of barbituric acid from an unsubstituted malonic or cyanoacetic derivative and introducing a second substituent into the barbituric acid molecule. For example, 50 pts. of diethyl monoethylmalonate are mixed with an equal weight of monochlorocrotonyl bromide, a solution of sodium ethoxide prepared from 6·25 pts. of sodium and 100 pts. of absolute alcohol is added, and the mixture boiled under reflux until neutral. The alcohol is removed by distillation and the residue is washed and distilled. *Diethyl ethylmonochlorocrotonylmalonate* passes over at 154—157°/18 mm. 100 pts. of this product are mixed with 32·5 pts. of carbamide in a solution of sodium ethoxide prepared from 20·8 pts. of sodium and 330 pts. of absolute alcohol, and the mixture is boiled under reflux for several hours, poured into water and acidified, when *CC-ethylmonochlorocrotonylbarbituric acid*, m.p. 162°, separates. *CC-di-(chlorocrotonylbarbituric acid* has m.p. 210—212° and *CC-phenylmonochlorocrotonylbarbituric acid* has m.p. 240—242°. T. S. Wheeler.

Dental filling material. P. W. Kruger (U.S.P. 1,584,560, 11.5.26. Appl., 19.6.24).—Kaolin is mixed with a solution of magnesium chloride or nitrate or of a mixture of these salts and the whole ignited, when a mixture of kaolin (65%) and magnesium oxychloride or oxide or both is obtained. A mixture of eugenol or guaiacol with 1 or 2 pts. of this powder sets rapidly to a hard germicidal cement suitable as a dental filling material. T. S. Wheeler.

Process for preparing vaccines. F. M. WOOD (U.S.P. 1,584,583, 11.5.26. Appl., 29.2.24).—Micro-organisms are treated with dilute alkali for periods of 3 min. to 48 hrs. at $-4°$ to 100° until the capsules present are disrupted. In certain cases dilute acetic acid may be used. If the capsule is of a fatty or waxy nature treatment with 40% formaldehyde followed by extraction with acetone is preferred. The suspension is treated in slightly alkaline solution with trypsin for 5—8 min. to digest the toxic proteins present, and then mixed with sodium oleate solution, which flocculates the capsules and with 0·5% of phenol to sterilise the mixture. After centrifuging for 2 min. at 1000 r.p.m. the liquid containing pure bacterial protein is decanted from the capsules and is standardised and rendered isotonic to normal saline.

T. S. WHEELER.

Purifying and deodorising *iso*propyl alcohol. M. D. MANN, JUN., and R. B. LEBO, Assrs. to S. B. HUNT (U.S.P. 1,585,042, 18.5.26. Appl., 27.10.21).—Crude *iso*propyl alcohol from the propylene present in oil-still gases is mixed with mercuric or zinc chloride (1—7 lb. per 1000 galls.) and distilled after 12 hrs. It is of advantage before distillation to treat the alcohol with about 8 galls. of caustic soda solution (*d* 1·11) or 5—10 lb. of bleaching powder per 1000 galls. of alcohol. The product on distillation is free from any foreign odour. Amyl alcohol may be similarly purified. T. S. WHEELER.

Process of making crotonaldehyde. C. J. HERRLY, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,585,096, 18.5.26. Appl., 22.5.24).—Aldol is prepared out of contact with oxygen in an atmosphere of an inert gas such as nitrogen and is heated at 165° in a current of live steam so that the crotonaldehyde is removed as formed and is condensed out of contact with oxygen. Fresh aldol is added to the heated liquid to replace that removed. The yield is good, and the crotonaldehyde obtained, owing to its freedom from impurities, gives good yields of butaldehyde and butyl alcohol when hydrogenated. T. S. WHEELER.

Oxygen compounds of terpene. A. S. RAMAGE, Assr. to OZONID CORP. (U.S.P. 1,585,602, 18.5.26. Appl., 6.4.23; cf. E.P. 4200 of 1901 and U.S.P. 1,097,939—40, and 1,098,356; B., 1902, 188; 1914, 844).—Ozonised air is passed through eucalyptus oil at 80° and the unstable terpene peroxide $C_{10}H_{18}O_2$ which is formed is removed by the current of air and mixed at 80° with excess of ozonised air when a more highly oxidised compound with the formula $C_{10}H_{18}O_3$ is produced. This substance forms a colourless liquid which in contact with water liberates active oxygen over a period of 36—48 hrs.

T. S. WHEELER.

Preparation of substituted ketopolyhydronaphthalenes. J. D. RIEDEL A.-G. (G.P. 422,036, 30.9.23).—Ketopolyhydronaphthalenes are condensed with aldehydes and the unsaturated $\alpha\beta$-ketones produced are caused to take up 1 mol. H_2 by means of a suitable reducing agent and form saturated ketones. Both classes of substances act on the muscular system, are slowly absorbed, and can be used as a remedy for worms. The following examples are given: *benzylidene-α-tetrahydronaphthalene ketone*, m.p. 108—110°, pale green plates, is prepared by the action of benzaldehyde and a solution of sodium methoxide on α-tetrahydronaphthalene ketone and on reduction with hydrogen in acetic acid solution in the presence of platinum black is converted into *benzyl-α-tetrahydronaphthalene ketone* m.p. 55—56°; *benzylidene-*ar*-methoxy-α-tetrahydronaphthalene ketone*, pale rose, m.p. 99—101°, by the action of benzaldehyde on *ar*-methoxy-α-tetrahydronaphthalene ketone, giving on reduction, *benzyl-*ar*-methoxy-α-tetrahydronaphthalene ketone*, m.p. 53—55°; o-*hydroxybenzylidene-β-decahydronaphthalene ketone*, yellow, m.p. 166°, is obtained by condensing β-decahydronaphthalene ketone with salicylaldehyde in alcoholic solution in the presence of sodium hydroxide (50%), and on reduction with zinc dust in acetic acid solution gives o-*hydroxybenzyl-β-decahydronaphthalene ketone*, m.p. 110°. A. COULTHARD.

Manufacture of methyl alcohol by the hydrolysis of methyl chloride. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of O. NICODEMUS and O. WULFF (G.P. 421,789, 27.7.20).—By using a catalyst, preferably a salt of copper, with the hydrolysing agent, the rate of hydrolysis of methyl chloride can be raised 4—5 times, and the reaction carried out at lower temperatures and pressures. For example, a mixture of water, calcium oxide, and a small quantity of cuprous chloride is heated to 120° in a rotating vessel. The pressure rises to 2 atm. Methyl chloride is forced in gradually, causing a final rise to 7—8 atm., the reaction being complete in about $1\frac{1}{2}$ hrs. At 140°, the pressure is about 10 atm. and the reaction ended in $\frac{1}{2}$ to $\frac{3}{4}$ hr. The yield amounts to 95%. A. COULTHARD.

Production of methyl chloride solutions. HOLZVERKOHLUNGS-IND. A.-G. (G.P. 425,610, 2.8.22; Austr. P. 99,679, 12.7.23).—After hydrochloric acid and higher chlorination products have been removed from the mixture formed on chlorinating methane, the methyl chloride remaining is taken up by methyl alcohol, preferably under moderately increased pressure or cooling or by the use of both. For example, the reaction product obtained by chlorinating methane is washed free from hydrochloric acid by water, and dichloromethane, chloroform, and carbon tetrachloride are removed by cooling. It is then led into methyl alcohol at 0—5° under 2—4 atm. pressure giving a mixture of about 50% each of methyl chloride and methyl alcohol. The residual methane can be led back to the chlorinating vessel. The 50% mixture can be used as a methylating agent or hydrolysed to methyl alcohol by means of alkali.

A. COULTHARD.

Manufacture of acetaldehyde from ethyl alcohol. HOLZVERKOHLUNGS-IND. A.-G. (G.P. 422,729, 19.12.23).—Alcohol vapour and oxidising gases such as air, are led over contact substances

containing silver in the form of gauze. Compared with the use of silver deposited on asbestos, the yield of aldehyde is raised from 70·4 to 93·6% and the life of the catalyst is prolonged. The reaction is not so violent, and only one contact tube need be used. For example, a 60 mm. length of a quartz tube of 18 mm. diam. is filled with discs of silver gauze lying one over the other. About 20 g. of alcohol and 25 litres of air are led through in 1 hr. A short heating raises the catalyst to a visible red heat, after which the reaction proceeds without further external heating.

A. Coulthard.

Preparation of aminoketones. C. Mannich (G.P. 422,916, 26.3.22. Addn. to 422,098; B., 1926, 514).—Secondary amines such as piperidine or hydrogenated quinoline are used instead of the mono- and di-alkylamines specified in the chief patent. For example, piperidinomethyl*cyclo*hexanone hydrochloride, m.p. 161°, is prepared from piperidine hydrochloride, formaldehyde (30% solution), and *cyclo*hexanone; the free base b.p. 145°/20 mm., has an amine-like odour. Piperidinomethyl*cyclo*pentanone hydrochloride, m.p. 145°, is similarly prepared from *cyclo*pentanone. Condensation of tetrahydro*iso*quinoline hydrochloride with formaldehyde and *cyclo*hexanone gives tetrahydro*iso*quinolylmethyl*cyclo*hexanone hydrochloride, m.p. 161°. These aminoketones are used for the preparation of pharmaceutical products.

A. Coulthard.

Preparation of *C*-benzylnaphthols. Farbenfabr. vorm. F. Bayer & Co., Assees. of W. Kropp (G.P. 422,948, 7.3.24).—The yields of benzylnaphthols are considerably improved if benzyl halides are allowed to react with naphthols in the presence of salts of weak acids capable of neutralising the hydrogen chloride liberated, instead of using alkali compounds of the naphthols. The formation of by-products such as naphthyl benzyl ether, dibenzyl ether, and benzyl alcohol is at the same time diminished. The reaction may take place in aqueous solution or in an organic solvent, and catalysts such as the salts of iron, copper, or zinc may be added. The benzyl naphthols are used as a specific for parasites in the bowel. α-Benzyl-β-naphthol m.p. 115°, is obtained by the action of benzyl chloride on an aqueous solution of β-naphthol in the presence of sodium or calcium carbonate, or borax, or in a solution of xylene in the presence of sodium carbonate and ferric chloride. 4-Benzyl-α-naphthol, m.p. 124°, is similarly prepared from α-naphthol and benzyl chloride in aqueous solution in the presence of sodium carbonate.

A. Coulthard.

Preparation of a derivative of 1-phenyl-2:3-dimethyl-5-pyrazolone. Farbw. vorm. Meister, Lucius, & Brüning, Assees. of F. Stolz and K. Böttcher (G.P. 423,028, 16.5.23).—4-Methylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone is treated with a benzylating agent or 4-benzylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone with a methylating agent. For example, a solution of 4-methylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone, m.p. 60°, in benzene is treated with benzyl bromide or a solution of 4-benzylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone in benzene is heated with methyl iodide in a closed vessel for several hours at 100°. The product, *4-benzylmethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone*, m.p. 68—70°, crystals, gives a violet colour with ferric chloride in hydrochloric acid solution, is very slightly toxic, and has a strong antipyretic action. 4-Benzylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone m.p. 73°, crystals, giving a reddish-brown colour with ferric chloride in dilute alcohol, is obtained by the reduction of the corresponding benzylidene compound with sodium amalgam.

A. Coulthard.

Preparation of piperazyltheobromine. E. A. O. Viel (F.P. 584,735, 17.10.23).—*Piperazyltheobromine*, m.p. 247°, is prepared by boiling for several hours aqueous solutions of piperazine containing halogen substitution products of theobromine.

L. A. Coles.

Purification of phenolphthalein (U.S.P. 1,583,372).—See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Spectral sensitivity of silver iodide and mixtures of silver iodide and silver bromide. E. Huse and C. E. Meulendyke (Phot. J., 1926, **66**, 306—309).—With silver bromide emulsions containing silver iodide, both the light absorption and spectral sensitivity show a progressive shift towards longer wave-lengths with increasing percentages of silver iodide up to 30—40%. At this point, the emulsions are almost orange in colour, compared with the normal clear lemon-yellow. At a point between 30 and 40% of iodide, *X*-ray analysis shows that iodide begins to crystallise out as separate crystals of silver iodide, instead of entering the cubic bromide lattice (cf. Wilsey, A., 1926, 113). A wedge spectrum photographed on a wet collodion plate shows a sharp absorption edge at 440 μμ. A pure silver iodide-gelatin emulsion, with physical development, gives exactly the same spectrum. In a series of silver bromide emulsions containing progressively increasing amounts of iodide, and using physical development, the sharp sensitiveness edge at 440 μμ was first observed with 32% of iodide; it was quite lacking with 30%, and very clear with 35%. It is concluded that in emulsions of the type used, silver iodide separates out as definite crystals, apart from the silver bromide, when the iodide content reaches 32%. W. Clark.

Comparative tables of spectral sensitivity of silver bromide, iodide, and chloride, and the action of the chief colour sensitisers. J. M. Eder (Z. wiss. Phot., 1926, **24**, 139—144).—Spectral sensitivity data concerning silver bromide–gelatin plates, silver bromide–collodion, wet silver iodide–collodion with ferrous sulphate development, silver chloride with chemical development, and a number of sensitising dyes on silver bromide, are tabulated. Exposures were to the solar spectrum. W. Clark.

Relation between colour sensitiveness and the gradation given by a photographic plate. T. T. BAKER and W. A. BALMAIN (Phot. J., 1926, **66**, 299—306).—The "gamma infinity" of a pure silver bromide–gelatin emulsion is constant for exposure to wave-lengths 3500, 4250, and 4750. A corresponding emulsion containing 2% of silver iodide shows the same constant γ_∞ for the ultra-violet and violet, and a decrease in the blue-green. An erythrosin-sensitised plate shows a considerable rise of γ_∞ in the greenish-yellow. Panchromatised pure silver bromide plates gave two distinct groups of γ_∞: a high constant value for the ultra-violet to middle green region, *i.e.*, the region to which normal silver bromide is sensitive, and a lower constant value for the middle green to red region, *i.e.*, that of sensitiveness added by the dye. Panchromatised iodo–bromide emulsion showed a linear and steady increase in γ_∞ from λ3500 to λ4600, and for higher wave-lengths a constant value. In colour sensitising it is supposed that the "sensitive centres" of the grains are made light-absorbent by reaction with the dye rather than the grains themselves. The change in general shape of the characteristic curve according to whether light of short or long wave-length is used in exposure, indicates that in colour-sensitising the sensitivity distribution among the grains is altered. In general, the gamma of dyed colour-sensitive silver bromide is different from that of the undyed salt, but only when exposed to light of the new spectral region to which the dye has sensitised it.

W. CLARK.

Action of light on silver bromide emulsions, and the rôle of the sensitising nuclei. F. C. TOY (Brit. J. Phot., 1926, **73**, 295—298).—Recent publications of Sheppard (B., 1925, 785; 1926, 219) are criticised. The fact that certain theoretical equations deduced by Silberstein do not hold experimentally is not evidence against the Einstein photochemical principle of the action of light on silver bromide. Sheppard's argument that the sensitive nuclei in emulsions act as general sensitisers and not as optical sensitisers is supported by reference to further work, but his statement that the nuclei cannot function as catalysts for the photochemical decomposition of silver halide, in the sense of increasing the amount decomposed per unit energy, is criticised. W. CLARK.

Desensitisation of colour-sensitive plates. A. HÜBL (Z. wiss. Phot., 1926, **24**, 133—139).—Desensitising power is not related to the colour of the desensitising dye. Colour-sensitivity of a plate is relatively more decreased by desensitising dyes than is blue-sensitivity, and with Pinacyanol plates, the relative effect increases in the order Phenosafranine, Pinakryptol Green, Pinakryptol Yellow. The facts agree with the view of Kögel and Steigmann (Phot. Ind., 1925, 1143) that sensitising dyes are so light-sensitive that in light of long wave-length they undergo a direct chemical change which results in developability of the neighbouring silver bromide. Desensitisers decrease the sensitivity of the sensitising dye as well as of the silver bromide. The rate of bleaching of Pinachrome solution in sunlight is decreased if Pinakryptol Yellow is added. Silver bromide stained with Pinachrome is stable to light in presence of Pinakryptol Yellow. Desensitisation of silver bromide and of its sensitising dye are independent, and different desensitisers which affect blue sensitivity equally may have varying effects on colour sensitivity. With Pinachrome Violet-sensitised plates and Scarlet N, blue and yellow sensitivity are depressed equally. Blue sensitivity is never decreased relatively more than colour sensitivity. In dilute solutions of desensitisers, decrease of sensitivity of both silver bromide and sensitising dye is proportional to the concentration of the desensitiser. W. CLARK.

Silver acetylide emulsions. G. KÖGEL (Phot. Ind., 1926, 582—583).—Silver acetylide made from neutral solutions is only slightly sensitive to light, and can be kept for two years in closed flasks. When treated with ammonia a complex is formed, which is sensitive. Silver acetylide forms highly sensitive complexes with other compounds, *e.g.*, thiocarbamide. On exposure such complexes are at first very sensitive, but the rate of darkening soon decreases owing to shielding by the liberated silver. Latent images cannot be developed on these substances, since the whole surface is reduced. Print-out images on silver acetylide-ammonia show greater contrast than silver halide images, but they are difficult to fix. W. CLARK.

See also A., June, 585, **Photochemical properties of chromates** (PLOTNIKOV and KARSULIN); **Photochemistry of silver chloride, bromide, and thiocyanate** (SCHWARZ and DIEFENBACHER). 590, **Electrometric titration of halides** (CLARK).

XXII.—EXPLOSIVES; MATCHES.

Calculations for the regeneration of nitrating acids. H. V. HANSEN (Ind. Eng. Chem., 1926, **18**, 598—601).—A graphical method is developed by which the amounts of sulphuric and nitric acids required to fortify used nitrating acid to any given specification can be ascertained, the method being easily adjustable to variations in composition of the fortifying acids and the regenerated nitrating acid required. B. W. CLARKE.

PATENTS.

[Flameless] propellent powder. C. R. FRANKLIN (U.S.P. 1,582,256, 27.4.26. Appl., 8.10.23. Free of use in United States).—Nitrocellulose (83·24%) containing 12·6% N is saturated with alcohol, incorporated with nitroguanidine (9·84%), a small quantity of an oxidising agent such as barium nitrate (4·48%) is added, and then a stabiliser, *e.g.*, diphenylamine (0·44%) dissolved in sufficient ether to give with the alcohol present a suitable solvent for the nitrocellulose. After mixing and forming into grains, the product is

heated at 40—50° for 6 days in a closed vessel and dried at 55°. The grains are then coated at 40° in a tumbling machine with a deterrent such as 1:2:4-dinitrotoluene (1·975%) and glazed with graphite (0·025%). T. S. Wheeler.

Nitroglucoside explosive. R. C. Moran, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,583,895, 11.5.26. Appl., 23.1.23).—Glucosides formed from the lower aliphatic alcohols and glycols and sugars such as dextrose or mannose may be nitrated under the same conditions as glycerin to give cheap stable nitroglucosides of low freezing point which can be used to replace a portion or all of the nitroglycerin in dynamite. They are good gelatinisers of nitrocellulose. Mixtures of nitroglycerin and nitroglucosides may be prepared by nitrating a mixture of glycerin and the glucoside. T. S. Wheeler.

Production of primers. Sprengstoffwerke R. Nahnsen & Co. A.-G. and G. Pyl (G.P. 423,176, 8.3.25).—Priming materials, suitable for replacing mercury fulminate in fuses, detonators, and caps, are prepared with acetone peroxide as main or secondary constituent. W. T. K. Braunholtz.

Production of explosives. Norddeutsche Torf-Kokerei A.-G. (G.P. 424,379, 16.10.24).—The efficiency of black powder and other explosives is increased by the use of peat flour, prepared by suddenly subjecting the peat mass as usually obtained to temperatures above 100°, carefully drying the product, and reducing it to flour. W. T. K. Braunholtz.

Removal of nitration acids from nitrocellulose. E. Pungs (G.P. 424,941, 19.4.23).—Nitration acids adhering to nitrocellulose after centrifuging are rapidly and completely removed by placing the material in a cylinder, covering with water, and drawing the latter with the aid of diminished pressure through the nitrocellulose into a collecting vessel. W. T. K. Braunholtz.

Machines for rolling explosives or other similar gelatinous substance. Greenwood & Batley, Ltd., and J. H. Barker (E.P. 252,048, 14.1 and 14.10.25).

XXIII.—SANITATION; WATER PURIFICATION.

Ammonia and nitrate content of lake waters. B. P. Domogalla, E. B. Fred, and W. H. Peterson (J. Amer. Water Works Assoc., 1926, **15**, 369—385).—Little change is observed in the soluble nitrogen content of the deep thermally-stratified lake waters of Wisconsin from autumn until midwinter, when sudden increases occur; the ammonia and nitrate contents reach a maximum in March. The increases are noticeable first in the bottom waters, later at various depths, and finally at the surface. During this period the oxygen of the lower strata decreases but does not entirely disappear. During the spring and autumn turnovers the soluble nitrogen content is uniform. In the summer, when stratification again takes place, the ammonia content again increases in the lower layers but the nitrate content decreases and, usually, the oxygen from the lower layers rapidly disappears. Bacteriological studies indicate that the seasonal fluctuations in the ammonia, nitrate, and oxygen content are due almost entirely to a seasonal variation in the bacterial flora of the waters, which is most marked in the lower strata. In general, an increase in the number of bacteria is followed by an increase in ammonia content. In winter, ammonifiers and nitrifiers are most active and in summer ammonifiers and nitrate reducers are predominant. W. T. Lockett.

Modification of the Gillespie approximate method of determining hydrogen-ion concentration [of water]. J. McCrae (Analyst, 1926, **51**, 287—290).—The apparatus is devised to avoid a large number of volume and drop measurements, or if not less than 300 c.c. of water are available, of any volume or other measurements. It consists of two right-angled triangular glass cells, placed together to form a divided rectangular cell, and each filled with the water under investigation mixed with a sufficient quantity of appropriate indicator solution, in one cell with the addition of a few drops of sodium hydroxide solution and in the other of acid. Over these cells is placed a cover fitted with lateral slots, and on the top of this is a small rectangular cell containing the water under examination. This cell is fixed on a carrier which enables it to be moved along the cover until the tint of the contained water against a white background matches the colour below one of the slots in the cover. A table is given of the p_H values corresponding to the different slots in the apparatus described, for seven indicators. Directions are given for working with small samples of water. D. G. Hewer.

Free chlorine in chlorinated tank effluents. W. V. D. Tiedeman (J. Amer. Water Works Assoc., 1926, **15**, 391—399).—The *o*-tolidine test for excess chlorine is applicable to sewage tank effluents when 1-oz. bottles are substituted for Nessler tubes, the amount of *o*-tolidine increased to 4 c.c. per 100 c.c. sample, and allowance made for turbidity. The results obtained on fresh sewage by starchi–odide titration using acetic acid are at variance with those obtained by the *o*-tolidine test, the excess chlorine indicated by the starch–iodide test being always much higher than that indicated by *o*-tolidine. Good bacterial removal and reduction of *B. coli* can be obtained in chlorinated effluents of tanks treating domestic sewage when the *o*-tolidine test indicates a slight excess of chlorine. W. T. Lockett.

Determination of dissolved oxygen in effluents. E. R. Trotman (J.S.C.I., 1926, **45**, 110 T; cf. Friend, A., 1925, ii, 326).—Winkler's method of determining dissolved oxygen is carried out in a 260-c.c. bottle fitted with a rubber bung, through which passes the stem of a tap funnel and a side

tube with a tap. The stem of the funnel is drawn out to a fairly fine tube, and passes to the bottom of the bottle. 10 c.c. of light petroleum are poured into the bottle, which is then filled to the 250-c.c. mark with the water. The reagents are added down the tap funnel and washed down with petroleum, excess of the latter being displaced through the side tube. Introduction of air is thus avoided.

A. B. Manning.

Determination of strychnine in poisoned grains. J. W. Elmore (J. Assoc. Off. Agric. Chem., 1926, 9, 224—230).—In connexion with the determination of strychnine in poison baits used in rodent control, it was found that extraction with kerosene gave lower results than a Soxhlet extraction with chloroform followed by precipitation of the alkaloid as picrate. As the latter method also gives low results, a satisfactory method of extraction based on Allen's method for nux vomica (Commercial Organic Analysis, 1912, 6, 469) has been worked out, the strychnine being extracted with a mixture of ether and chloroform (2:1) and finally weighed as picrate.

C. O. Harvey.

See also A., June, 591, **Winkler's method for determination of dissolved oxygen and its use in presence of oxidisable substances** (Alsterberg). 637, **Chlorine gas treatment for respiratory diseases** (Gilchrist).

Patents.

Process of softening water. M. F. Newman, Assr. to W. B. Scaife & Sons Co. (U.S.P. 1,572,944, 16.2.26. Appl., 19.4.24).—Feed water for boilers is reduced to minimum hardness with relatively low alkalinity by a standard precipitation process using lime and soda ash or other reagents. The water is then treated with an acid sulphate, *e.g.*, sodium bisulphate, to reduce further the alkalinity, or with a suitable reagent, *e.g.*, tannic acid, to increase the solubility of the residual calcium, or with both reagents.

W. T. Lockett.

Purifying boiler-feed water. O. Günther (U.S.P. 1,579,444, 6.4.26. Appl., 17.7.23).—When the boiler is delivering steam for consumption, the feed water is preheated so that the deposit will form before the water passes into the boiler, and it is then led into the water space through a sludge separator. When the boiler is not producing steam for consumption, the cold water is led into a chamber in the steam space where it is heated to the temperature of the steam and forms scale, after which it passes to the main body of water.

E. S. Kreis.

Preparation of a base-exchanging material from clay for softening water. Permutit A.-G. (G.P. 426,083, 12.10.19. Addn. to 423,224; B., 1926, 470).—The clay, before granulating and firing, is enriched with substances which can interchange bases. After firing, while still hot, it is treated with solutions of alkalis. The base-exchanging capability of the clay is increased.

A. Coulthard.

Biological purification of sewage with activated sludge. K. Imhoff (G.P. 426,766, 19.9.24).—The sewage in the aerating chamber is given a spiral motion by spraying the surface with purified effluent and separating the sludge from the sewage as it comes from the aerating chamber by causing it to flow into a chamber with a sloping bottom, on which the sludge deposits and whence it slides back into the aerating chamber.

E. S. Kreis.

Process for producing a liquid insecticide containing the effective ingredients of *Derris* species. Zaidan Hojin Rikagaku Kenkyujo (E.P. 239,483, 24.4.25. Conv., 8.9.24).—Material containing the active ingredient of derris, obtained, *e.g.*, by extraction of the root with an organic solvent and concentration of the extract, is mixed with one or more oils, fats, waxes, resins, higher alcohols, hydrocarbons, or fatty acids (excluding Turkey-red oil), which are free from water and alkali. This mixture is stable and retains insecticidal properties for a long time; for use, it is added to an aqueous solution of soap, glue, or other material giving a colloidal aqueous solution and is then suitably diluted with water.

C. T. Gimingham.

Destruction of vermin. H. E. Potts. From Chem. Fabr. H. Stoltzenberg (E.P. 231,497, 25.3.25).—Vermin in a closed space are exposed to the action of hydrocyanic acid generated by the interaction of additive compounds of the acid and anhydrous metal chlorides, such as aluminium or ferric chloride (cf. E.P. 250,453, p. 583) with a limited quantity of water, namely, about 90 to 120% of the weight of the additive compound.

T. H. Pope.

Purifying liquids [water]. M. D. Avery (E.P. 251,136, 17.6.25).—See U.S.P. 1,554,052; B., 1925, 780.

Treating [base-exchanging] minerals. E. B. Higgins, Assr. to United Water Softeners, Ltd. (U.S.P. 1,586,365, 25.5.26. Appl., 20.6.25).—See E.P. 244,506; B., 1926, 174.

Clarifying and decolorising the outflow water from works. G. Ullmann (E.P. 241,179, 24.9.25. Conv., 10.10.24).—See Austr. P. 100,735; B., 1926, 470.

Apparatus for treating liquids. [Removing gases from boiler-feed water.] Griscom-Russell Co., Assees. of R. C. Jones (E.P. 234,448, 2.3.25. Conv., 24.5.24).

[Visual indicator of flow of water through] pressure filters. D. McG. Stewart (E.P. 253,458, 13.2.26).

Filtering waste water. E. Geiger (E.P. 251,474, 4.7.25).

Soluble oils (U.S.P. 1,582,257).—See II.

Salts of *N*-halogen-substituted arylsulphonamides (G.P. 422,076).—See XX.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUGUST 6, 1926.

I.—GENERAL; PLANT; MACHINERY.

Distillation studies. E. H. LESLIE and J. C. GENIESSE (Ind. Eng. Chem., 1926, 18, 590—596).—Chloroform–toluene mixtures were distilled in an apparatus fitted with a specially designed laboratory fractionating column. Thermal efficiencies of all parts of the apparatus were determined, and the results compared with those of a hypothetical column with an infinite number of equilibrium units. The data obtained indicate that reflux ratio may be without influence on the effectiveness of a well-designed fractionating column, while vapour velocity can be neglected as a variable factor in column design and operation provided it is not high enough to cause excessive entrainment. The effectiveness of column packings varied inversely with the diameter. Charts are shown representing the relationship of reflux ratio, number of equilibrium units, and change in composition of chloroform–toluene mixtures. B. W. CLARKE.

Dialysis, filtration, percolation. Pore diameter of separating surfaces. A. MÜLHAUS (Kolloid-Z., 1926, 39, 37—40).—A discussion of various filtering and sieving surfaces, special attention being devoted to wire and silk gauzes and the discrepancies which sometimes occur between the actual pore diameter and that calculated from the number of filaments per centimetre as stated by the manufacturer.
N. H. HARTSHORNE.

Some little-understood factors affecting lubrication. E. G. GILSON (Ind. Eng. Chem., 1926, 18, 467—470).—Changes in room temperature were found to cause a difference of 20% in the friction of a bearing. An apparatus was constructed in which a ring was rotated with a clearance of 0·013 in. within a bowl of shafting steel, and the friction curve for a bronze ring was found to be considerably higher than for copper. When run in an atmosphere of hydrogen the friction increased in the case of mineral oils, but decreased with castor and sperm oils. The oils blackened and sludged in air, but not in hydrogen. Minimum friction is accompanied by a reaction between the oil and metal dependent upon the presence of oxygen or air. In another apparatus runs were made *in vacuo*, and the friction was found to be greater than in air. The friction in oxygen was less than in air. After running in air or oxygen the friction gradually increases on running in hydrogen or *in vacuo*, but rapidly decreases on running again in air or oxygen. The addition of moisture to the hydrogen causes diminution of the friction. H. MOORE.

Surface action and fluid film lubrication. A. E. BECKER (Ind. Eng. Chem., 1926, 18, 471—477).—An apparatus was devised for measuring the thickness of an oil film by the capacity of the insulated surfaces of a bearing as an electric condenser. Polished flat thrust bearings ½ in. in diameter were used, the lower one being mounted in a bakelite–micarta block, and the upper one attached to a revolving spindle, which could be weighted as desired. The bearing surfaces were connected electrically to a Wheatstone bridge including an audio-oscillator and telephone receiver, the condenser capacity of the bearing being balanced by an adjustable capacity. Cast iron on cast iron was used as a bearing in which the lubricant adheres to both surfaces, magnesium on magnesium as a bearing in which the lubricant adheres to neither surface, and cast iron on magnesium for the case in which the lubricant adheres to one surface only. The bearing temperature was kept at 21·1°; the speeds varied from 1395 to 400 r.p.m., and the loads were varied. A number of graphs show the results of determinations of thickness of films and the effects of variations of temperature, pressure, and speed. A general equation for film thickness has been developed. H. MOORE.

Laboratory muffle kiln. MERRITT.—See VIII.

PATENTS.

Pulverising or grinding mills. J. B. C. SCHERBAUM (E.P. 245,434, 15.12.25. Conv., 30.12.24).—A disc grinder is provided with rings of teeth of special form. B. M. VENABLES.

Tube and ball mills. P. V. A. A. HERBST (E.P. 251,564, 5.1.26. Conv., 29.8.25).—The shell or drum of a ball mill is supported on a hollow trunnion which communicates with the interior of the shell, is closed at its outer end and has side openings through which the material passes. A driving mechanism in line with the shaft is connected, flexibly if desired, with the trunnion.
S. BINNING.

Centrifugal separators. AKTIEBOLAGET SEPARATOR (E.P. 246,457, 6.1.26. Conv., 21.1.25).—The continuous discharge of a centrifugal separator passes, not into a casing surrounding the separator but through pipes into a closed tank which may be connected by one or more pipes to the casing of the centrifuge, thus providing a closed vapour circuit and easy access to collecting tank and centrifuge. S. BINNING.

Apparatus for obtaining intimate contact between gases and liquids. British Oxygen Co., Ltd., and C. R. Houseman (E.P. 251,331, 28.1.25).—The method described in E.P. 189,442 (cf. G.P. 381,444; B., 1924, 42) is improved by using elements in which the interstices or passages have a diameter smaller than 0·1 mm. An element composed of alternating corrugated and plain ribbons spirally wound is suitable. S. Binning.

Interchange of heat between liquids. O. Soderlund, T. Gram, and Techno-Chemical Laboratories, Ltd. (E.P. 251,344, 30.1.25).—In the interchange of heat between liquids by means of a gaseous carrier which conveys vapour of the hotter liquid to the cooler one, the amount of carrier in the mixture of vapour and carrier circulating between the hotter and the cooler chamber is varied so as to maintain a more or less constant difference in temperature between the carrier and the liquid throughout the cycle, the volume of carrier passing regions of a chamber at different temperatures being varied as the heat-carrying capacity of the carrier varies. The circulating mixture may be divided into several streams flowing from successively hotter regions of the hot chamber to slightly cooler regions in the cool chamber. S. Binning.

Recovery of volatile solvents. J. H. Brégeat (E.P. 251,492, 30.7.25).—The products of hydrogenation of alicyclic hydrocarbon compounds, such as hydroaromatic compounds, terpenes, pinene, and oil of turpentine, are used alone or mixed with other known absorbents, for the absorption of the vapours of volatile solvents. S. Binning.

Drying plant. H. Schultz (G.P. 426,829, 23.9.22).—The main pipe conveying air from the dryer back to the heater has two branches which are laid parallel to the main pipe and pointing in opposite directions. A valve slides across all three pipes. It consists of a flat plate with a perforation so placed that when the valve is depressed to the full extent the two branches are shut off and there is a through passage for the main. When the valve is elevated, the cross-sectional area of the main pipe is diminished and the inlet and outlet pipes are opened by an amount equal to the amount by which the cross-sectional area of the main has been diminished, so that the volume of air escaping from the system is exactly equal to the volume entering. E. S. Kreis.

Apparatus for chemical production and research. M. Brutzkus (U.S.P. 1,586,508, 25.5.26. Appl., 4.8.20. Conv., 4.8.19).—See E.P. 149,915; B., 1922, 87a.

Distillation. J. Schneible (U.S.P. 1,587,188, 1.6.26. Appl., 7.12.20).—See E.P. 182,832; B., 1922, 680a.

Heat exchangers. Griscom-Russell Co., Assees. of J. Price (E.P. 232,176, 31.1.25, and 235,147, 4.4.25. Conv., 12.4 and 3.6.24).

Apparatus for separating liquids [of different densities]. Pirbright Co., Ltd., and A. Beale (E.P. 252,077, 18.2.25).

Apparatus for separating solid substances from waste water and other liquids. W. E. Evans. From Geiger'sche Fabrik Ges. (E.P. 252,642, 16.1.26).

[Discharging device for] continuously operating shaft furnaces. E. Sobek (E.P. 253,391, 2.9.25).

Alloy for thermocouple casing (U.S.P. 1,581,380).—See X.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Oxygen bomb method for sulphur determination [in fuels]. M. J. Bradley, R. M. Corbin, and T. W. Floyd (Ind. Eng. Chem., 1926, **18**, 583—584).—Pure sulphur burned in an oxygen bomb forms an equilibrium mixture of sulphur dioxide and trioxide, the composition of which depends on the maximum temperature attained, rate of cooling, etc.; it is not materially affected by increasing the initial pressure of the oxygen to 50 atm. The addition of compounds which form water vapour and oxides of nitrogen during combustion promotes an activating atmosphere in the bomb resulting in complete oxidation to sulphur trioxide. The sulphur content of coals, crude petroleums, oils, tars, asphalts, pitch, petrol, etc., can be determined in this way by the addition of ammonium nitrate to the charge in the bomb. B. W. Clarke.

Low-temperature carbonisation of coal preliminary to coking. A. Thau (Fuel Econ., 1926, **1**, 367—369).—Recent investigations show that by the addition of pitch to non-caking coal prior to carbonisation a good coke can be produced. The best results are obtained when the pitch is added in the form of a solution. The binding action is entirely due to a particular extractable high-melting fraction of the pitch, the addition of 7—8% of which will suffice to make even lignite yield a hard coke. By removing the low-boiling pitch constituents from coal by low-temperature carbonisation coke formation at subsequent higher temperatures is greatly favoured. The product resulting from distillation of coal at a temperature below which the bituminous bonding constituents distil, is uncaked and can be carbonised either alone or mixed with untreated coal at a high temperature to give a hard metallurgical coke. S. Pexton.

Gasification and carbonisation of peat. G. Keppeler (Stahl u. Eisen, 1926, **46**, 631—635, 742—750).—The higher moors, by reason of the ground waters which permeate them, yield a purer peat. Figures are given relating to the use of peat producer-gas in various works. Any type of producer is suitable, but restriction of the blast

pressure and of the air-inlet orifice may be an advantage. The gas has a slightly higher calorific value than producer-gas from coal. Its uniformity of composition and its freedom from sulphur render it eminently suitable for metallurgical processes. Peat gave good results in a revolving producer when gasified with coal, and when it was used alone no trouble in the routine of works processes was experienced. The cost of producer-gas from peat is slightly less than from coal. Owing to the low ash-content of peat clinkering of the producer is but occasionally necessary. In the carbonisation of peat drying takes place up to 100°; from 100° to 200° more water is split off and carbon dioxide is evolved; above 200°, and markedly at 250°, the process becomes strongly exothermic, most of the tar is evolved, hydrogen, hydrocarbons, and carbon monoxide appear in increasing quantities, but much carbon dioxide still comes off; in the interval 350° to 480° the carbon dioxide gives place to combustible gases. The resulting coke possesses a low ignition-temperature and a high reactivity. Its low sulphur and phosphorus contents enhance its value for metallurgical purposes. A block of peat on carbonisation and gasification shrinks, but maintains its integrity to the end, except for some abrasion. Carbonisation processes using internal heating are highly efficient thermally, but the combustible gases are largely diluted, especially if there is much moisture in the peat. A reactive coke is obtained but gasification reactions may take place with attendant disadvantages. In the Domnick process both internal and external heating are used and a series of chambers are provided, one for each stage of the carbonisation process. The by-products, acetic acid, methyl alcohol, acetone, and ammonia, do not occur in sufficiently high concentration in the inevitably large quantity of liquor to repay present methods of recovery. R. A. A. TAYLOR.

Chemical reactions in the gas producer. J. A. GOFF (Ind. Eng. Chem., 1926, **18**, 585—588). —Assuming that producer-gas contains no free oxygen, and is in chemical equilibrium with the fuel (considered as pure carbon), and that just enough steam is introduced with the air to keep the temperature in the producer constant, the behaviour of the producer under varying conditions can be calculated from the laws of chemical equilibrium and thermodynamics, the fundamental equations involved being $C+O_2=CO_2$, and two of the equations $C+H_2O=H_2+CO$; $C+2H_2O=2H_2+CO_2$; $C+CO_2=2CO$; $CO+H_2O=CO_2+H_2$. Thus the nitrogen content of the producer-gas will increase with rise of temperature, since the higher the temperature the less steam is required to maintain the temperature constant. A maximum hydrogen content is obtained at about 950°, but this diminishes at higher temperatures, while the carbon monoxide content increases to a definite maximum, and then remains approximately constant. Above 1150°, carbon dioxide and water practically disappear, while below 700° steam or water vapour goes right through the producer as such without reacting at all. The efficiency of the producer increases up to about 1100°, but then decreases owing to sensible heat losses in the producer-gas. Approximately 0·4 lb. of steam should be used per 1 lb. of carbon for maximum efficiency under the conditions assumed.
B. W. CLARKE.

Combustion in gas engines. W. T. DAVID (Faraday Soc., June, 1926. Advance proof).—A study of recent work on gaseous explosions in closed vessels leads to the conclusion that incomplete combustion is an important factor in limiting the maximum pressure developed. In one instance, where the calculated pressure rise in the cylinder of a gas engine, assuming complete combustion and no heat loss, and taking account of the variation of the specific heats with temperature, was 460 lb. per sq. in., the rise actually observed was 320 lb. per sq. in. Allowing about 7% for heat loss, there remains a loss of 115 lb. per sq. in., which is attributed to incomplete combustion. The effect of composition of charge, turbulence, density, temperature, radiation from the walls (cf. B., 1925, 793), position of spark, shape of chamber, and time of ignition on the rate of combustion in gas engines is briefly discussed. A. B. MANNING.

Sulphur compounds of Kimmeridge shale oil. F. CHALLENGER, J. HASLAM, R. J. BRAMHALL, and J. WALKDEN (J. Inst. Petrol. Tech., 1926, **12**, 106—141; cf. B., 1925, 161).—The fraction of Kimmeridge shale oil volatile in steam (about 30%) was redistilled up to 180° at normal pressure and above 180° at reduced pressure, and the distillates were subjected to repeated fractionation giving 7 fractions as follows:—(1) up to 93°, (2) 109—117°, (3) 117—126°, (4) 132—140°, (5) 158—167°, (6) 105—115°/27 mm., (7) 115—140°/27 mm. In addition to thiophen and 2-methylthiophen in the first and second fractions (cf. B., 1925, 161), 2-ethylthiophen, 2:3-dimethylthiophen, and tetrahydrothiophen were isolated from fraction (4). Fraction (5) gave a compound believed to be a propyl-, a methylethyl-, or a trimethyl-derivative of thiophen. Naphthalene and thionaphthen were isolated from fraction (6). An extensive bibliography is appended.
S. BOWMAN.

Explosive reactions considered in reference to internal-combustion engines. D. CLERK (Faraday Soc., June, 1926. Advance proof).—A very brief survey of previous work on the calculation of the efficiency of internal-combustion engines.
A. B. MANNING.

Effect of metallic sols in delaying detonation in internal-combustion engines. C. J. SIMS and E. W. J. MARDLES (Faraday Soc., June, 1926. Advance proof).—The action of lead tetraethyl and similar organometallic compounds in preventing detonation is probably related to the ease with which they decompose on heating, yielding the metal in a very finely-divided and chemically active state. The metallic particles act as combustion centres,

causing ignition in the gas which otherwise would detonate on compression. Colloidal solutions of iron, lead, and nickel are just as effective as the organo-metallic compounds, sometimes slightly more so, in delaying detonation. With gold and carbon sols, however, negative results were obtained. Most of the metal sols were prepared by heating solutions of the carbonyls or ethyl-derivatives to a sufficiently high temperature, and preventing coagulation of the metal by addition of a protective colloid such as caoutchouc. The tests were made on a Ricardo variable compression engine, the degree of pinking being determined by ear. When different metallic compounds were compared, using the same concentration of metal, the most efficient was iron pentacarbonyl, which, at a concentration of 2 g. of metal per litre of petrol, gave an increase of 61% in the H.U.C. (highest useful compression) value.

A. B. MANNING.

Explosions in petrol engines. H. T. TIZARD (Faraday Soc., June, 1926. Advance proof).—Recent work on the maximum pressures produced when mixtures of carbon monoxide or hydrogen with air or oxygen are exploded in closed vessels enables a re-determination to be made of the mean specific heats of water vapour and carbon dioxide, and of the degree of dissociation of the latter, up to 2500° (cf. Tizard and Pye, Phil. Mag., 1922, 79). The re-calculated values are: C_v (50—2500°) of water vapour 11·2 cal. per mol., and of carbon dioxide 10·5 cal. per mol. These may be compared with Bjerrum's values (Z. physikal. Chem., 1912, 79, 513) of 9·7 and 10·9 cal. per mol., respectively. At 2650° carbon dioxide is dissociated to the extent of about 23%. According to the revised figures the ideal efficiencies calculated previously by Tizard and Pye are, if anything, too high, whereas the high thermal efficiencies actually observed indicate that the calculated values are too low. A possible explanation of the discrepancy is that the figures for the dissociation, though consistent among themselves, are too high, and either that equilibrium is not reached at 2500° before the gases start to cool rapidly, or, more probably, that large differences of temperature exist in the gas after explosion. The cause of detonation is discussed, it being assumed as a working hypothesis that the occurrence of detonation with a particular fuel is dependent on the temperature in the unburnt part of the mixture reaching a definite value characteristic of the fuel. It follows that anything which lowers the minimum ignition temperature ought to increase the tendency to detonation. The effect of increasing the pressure and of altering the concentration of the fuel in the gaseous mixture is considered from this point of view. With few exceptions the experimental results can be reconciled with the hypothesis. No satisfactory explanation of the action of such "anti-knock" substances as lead tetraethyl has yet been proposed. Some modification of Callendar's suggestion (cf. following abstract) that these high-boiling constituents form liquid drops on compression which are more reactive than the vapour, will probably prove to be the right explanation. It does not apply, however, when the hydrocarbon or other substance is easily volatile, the determining factor then being probably the attainment of a very high temperature in some portion of the gas during the explosion.

A. B. MANNING.

Dopes and detonation. [Motor fuels.] H. L. CALLENDAR, R. O. KING, and C. J. SIMS (Engineering, 1926, 121, 475—476, 509—511, 542—545, 575—576, 605—608).—After a brief statement of the general theory of the detonation of liquid fuels and a review of previous work, a nuclear theory of self-ignition is outlined. The nuclei of liquid heavy paraffins present in the compressed air-fuel mixture on ignition are thought to be the most usual cause of detonation with ordinary petrol. These heavy paraffins have the lowest ignition temperatures of all the constituents of the petrol. A certain amount of adsorption may take place on the carbon present in the cylinder. The organo-metallic anti-knock "dopes" appear to coat the nuclei with a film of metal and so check the tendency to detonate. Three types of detonation are distinguished, viz., the pressure-wave type in which the mixture is fired by a compression wave of sufficient intensity; the nuclear type, depending on the presence of particles of relatively low ingition temperature; and the molecular type in which the temperature of a completely gaseous mixture is raised sufficiently to induce direct molecular combination. The nuclear type is the most common in petrol engines. The anti-knock effect of carbon disulphide and the knock-inducing effect of ether are explained on the nuclear hypothesis. Experiments were made in a sealed tube to investigate the behaviour of organo-metallic "dopes" at high temperatures and pressures. Lead tetraethyl began to boil at 210°, and exploded at 215°; when the substance is used in minute quantities in petrol, this explosion would be enough to cover the surface of the nuclei with metallic lead, while it would not be sufficiently violent to pre-ignite the mixture. Lead tetraethyl when mixed with ether at 243° decomposed with formation of yellow clouds, probably lead oxide; this would explain the destruction of the anti-knock effect of lead tetraethyl by ether. "Ethyl fluid" (a mixture of lead tetraethyl and ethylene dibromide) showed separation of lead, which reacted later to form lead bromide. A mixture of lead tetraethyl and water decomposed at 193°, leaving a pellicle of lead at the surface of the water. Nickel carbonyl started to deposit nickel at 100° and was heated to 260° without explosion. The inferiority of nickel carbonyl compared with lead tetraethyl is thought to be due to its greater volatility. Other organic compounds were sought which would be expected, from their physical constants, to raise the ignition temperatures of the petrol nuclei; the criteria desired were solubility in petrol, high critical temperature, high ignition temperature, and b.p. about 200°. The aromatic amines were found to possess these properties. From an examination of the critical temperatures and pressures, the latent heat and specific heat of the higher paraffins likely to be present in petrol, it was shown that the presence of liquid drops in an engine cylinder at the time of

ignition was possible. Some engine experiments on detonation were made in a Ricardo variable-compression engine. With an addition of 0·2% of lead tetraethyl to the fuel a 33% increase in the highest useful compression ratio (H.U.C.R.) was obtained, whilst with an addition of 0·4% the increase was 50%. The greater the charge density, the greater is the effect of lead tetraethyl. The H.U.C.R. of hexane, octane, and nonane were found to be 4·8, 4·6, and 3·9, respectively. Arranged in order of decreasing anti-knock efficiency the order was alcohol, toluene, benzene for a 25% addition, and toluene, alcohol, benzene for a 100% addition. A large number of organic substances were tested for anti-knock properties in 5% solutions in petrol. Methylaniline possessed half the anti-knock efficiency of aniline, and dimethylaniline showed no anti-knock properties; the toluidines and xylidines arranged in descending order of efficiency were *meta*, *ortho*, *para*. For solutions of methylaniline and *m*-toluidine a maximum value of H.U.C.R. appears to be attained at 15% concentration. The distillation curves of standard fuels were not affected by the addition of 5 c.c. of lead tetraethyl per gallon. The calorific values of various aromatic amines were determined, and it was estimated that a 15% solution of an aromatic amine in a standard petrol would lower the calorific value 2%. W. N. Hoyte.

Retarding of ignition produced by substances known as anti-detonators. M. Dumanois (Compt. rend., 1926, 182, 1526—1528).—An experiment using a gas mixture containing 1/1000 part of lead tetraethyl has shown that premature explosion is perceptibly diminished by this substance. The so-called anti-detonators, therefore, play the part of retarders. J. Grant.

Regulations proposed following lead tetraethyl investigation (Ind. Eng. Chem., 1926, 18, 432—433).—The regulations follow the recommendations of the U.S. Surgeon General's Committee (B., 1926, 260). In manufacturing lead tetraethyl and its blending to make "ethyl fluid," for addition to gasoline, the workers shall be given sanitary instructions and submitted to periodical medical examination, records being kept. Efficient ventilation is to be provided, containers labelled, and cleansing and filling must take place in a closed system. A dye shall be added to the "ethyl fluid." The maximum amount to be added to the gasoline shall be 1/1260 of the commercial, or 1/1300 of the pure compound. It shall generally be done in tank car lots at distributing centres, which shall be registered, and be in the open air, or provided with adequate ventilation. Precautions must be taken against leaks or spilling. A group of at least forty men must be periodically medically examined, the results being reported and recorded. H. Moore.

Determination of unsaturated hydrocarbons. J. S. S. Brame (J. Inst. Petrol. Tech., 1926, 12, 221—222).—In continuation of previous work (B., 1926, 395) the effect on the aromatic hydrocarbons of the sulphuric acid used for the removal of unsaturated hydrocarbons has been determined. Acid of 85% concentration removed about 30% and 88% acid removed 50% of the aromatic hydrocarbons from a mixture of cracked spirit and toluene. S. Bowman.

Direct production of "end-boiling-point petrol" from heavy oils. G. Egloff and V. Henny (Petroleum, 1926, 22, 606—609).—By marketing cracked petrol in the state in which it leaves the cracking plant, polymerisation in further processes and the loss of anti-detonating hydrocarbons is avoided. The yields of the "no residue" process, which yields 53—78% of petrol, are compared with those of the residue process. The production of a Dubbs' plant in which the residue was continuously withdrawn, operating on Kevin-Sunburst fuel oil, was 50—61% of the raw material, and on another plant, operating on Kentucky fuel oil, 58—61%. Details are given of results with one passage of the raw material. Californian cracked distillates often contain 1% of sulphur, more difficult to remove than that in petrols from Mid-Continental oils. The oil of the Smackover field is difficult to refine, but produces 48·9—52·1% of petrol of good anti-detonating qualities. The method of simultaneous cracking and coke production requires but slight alteration in working conditions. Details are given of the production of "Navy end-point petrol" in a Californian plant with a yield of 58—66%. H. Moore.

Rôle of oiliness in industrial lubrication. W. C. Wilharm (Ind. Eng. Chem., 1926, 18, 463—467).—In the apparatus employed static friction, as a measure of oiliness, was ascertained by observation of the angle at which a slider moved on an inclined polished brass surface. The slider consisted of 3 balls 1 in. in diam. or 3 steel pegs 0·1 in. in diam. The surfaces were ground with fine emery and with alumina in a liquid of slight lubricating properties. With a Pennsylvanian lubricating oil the friction coefficient rose with increase of load up to 500 g., when it became constant at 0·176. The coefficient for a crude oil closely agreed with that of a refined oil prepared therefrom. In the tests the softer metal was cut, and the harder metal became coated with brass at the point of contact. It is probable that the polished surfaces still presented asperities, and the function of a good oily film is to prevent these interlocking. The friction is a function of the attraction of the metals, their tensile strength, and the internal friction of the lubricant. H. Moore.

Lubrication data from Co-operative Fuel Research [U.S. Bureau of Standards]. S. W. Sparrow and J. O. Eisinger (Ind. Eng. Chem., 1926, 18, 482—485).—Experiments were made on a 4-cylinder truck engine with $4\frac{3}{4}$ in. bore and 6 in. stroke. The dilution of a lubricant in the crank-case tends to attain an equilibrium value. Equilibrium oils, to which a diluent has been added to approximate to that found under crank-case conditions, have been used successfully, the added diluent being evaporated

as additional diluent accumulates. Crank-case dilution increases with decreased volatility of fuel, with low temperature of cooling water and cylinder wall, and with increase of engine load and of fuel-air ratio. Viscosity of lubricant does not affect the rate of dilution. High crank-case temperature promotes the elimination of the diluent. Piston and ring fit does not affect dilution greatly. Break-away torque is not affected by the viscosity of oil on the cylinder wall, but is decreased by "oiliness," as by the addition of lard oil. Corrosion of parts may be caused by the temperature of the cylinder walls being below the dew point of water. The most favourable conditions for dilution and corrosion are frequent starting of the engine without prolonged running to enable it to attain normal temperature.
H. Moore.

Lubricating value as related to certain physical and chemical properties of oils. L. W. Parsons and G. R. Taylor (Ind. Eng. Chem., 1926, **18**, 493—496).—Lubrication is mainly influenced by the ratio, viscosity × speed of rotation/pressure on bearing, and by the capacity of the oil to form an adsorbed film. In a graph of the coefficient of friction the curve rises slowly with this ratio on that side of the critical point which appertains to fluid film lubrication, whilst it rises abruptly on the side representing partial lubrication, where the film-forming properties are of primary importance. Oiliness is best tested by the Wilson and Barnard static friction test and the Deeley friction test. In turbine lubrication the first sludge formed is soluble in hot oil, but separates on cooling, while on further oxidation an insoluble sludge is formed. The film-forming qualities of turbine oil are of minor importance. For cutting oils oiliness is essential and straight fatty oils are the best. In automobile engines, pistons, piston rings and cylinder walls operate in the region of partial lubrication, as also the brake surfaces of Ford cars. Graphs representing the working of a 6-cylinder Continental motor show that oils change decidedly in emulsion test after running for 15 min. The A.S.T.M. pour point test does not show the relative tendencies of oils to flow under pressure at low temperatures, this being better tested on a modified Bingham plastometer. Paraffin-base oils at —18° are not viscous but plastic, with low rates of flow at low pressures, asphaltic oils showing higher rates of flow. Gears which operate in the region of partial lubrication require greases of high viscosity and oiliness. These may be tested in the plastometer using a slightly larger capillary. H. Moore.

Power and the viscosity of oil. W. F. Parish (Ind. Eng. Chem., 1926, **18**, 525—526).—Tests were carried out in textile mills in Germany to compare Russian and American lubricating oils. While in some cases the viscosity of the oils was the same, the American oil showed a power saving of 15%, and while in laboratory tests oils of the same viscosity show the same coefficient of friction, more lengthy trials on machinery show an advantage for the more homogeneous oil against the blended one. Blended Russian oils showed greater change in colour, acidity, and viscosity than the American oils after running for four weeks. H. Moore.

Possible tests for the oiliness of oils. A. H. and H. Gill (Ind. Eng. Chem., 1926, **18**, 527).—Perrott's method for measuring the "length" of carbon blacks was used to determine the oiliness of oils. In place of carbon, infusorial earth ignited to burn out organic matter was used. The deflection of the oil was first measured in degrees MacMichael at speeds varying from 5 to 30 r.p.m. at 25°, and the results compared with that of a mixture of 10 g. of earth with 100 c.c. of oil. The "spread," or difference between the lines obtained with the oil and with the mixture, was converted to centipoises, using glycerin solutions of known gravities as standards. Castor oil and lard, which are known to be better lubricants than petroleum oils, give less "spread" than the latter, and the same may be said of porpoise jaw, lard, and rapeseed oils. H. Moore.

Distillation of Mexican crude oil emulsions and simultaneous preparation of asphalt emulsions. P. J. de Kadt (J. Inst. Petrol. Tech., 1926, **12**, 217—220).—Experiments are described for the conversion of typical asphaltic crude oil emulsions of the water-in-oil type into the oil-in-water type suitable for road use and roofing purposes. The inversion was readily effected by the addition of potassium oleate, but the resulting product was unsuitable on account of the gasoline and kerosene present. This difficulty was overcome by combining distillation with the treatment, but under these conditions the hydrophile tendency of the oleate was too strong and frothing occurred during distillation. Using a rosin soap, however, the inversion could be controlled so that distillation of the light products proceeded quietly. During this process the emulsion appears to be in a condition intermediate between the water-in-oil and oil-in-water types, and the residue is converted into an oil-in-water emulsion by the addition of sodium oleate. Generally 1—1·5% of rosin oil is required for the first stage and 0·5—1% of oleate for the second stage. S. Bowman.

Factors affecting lubrication. Gilson.—See I.

Surface action and fluid film lubrication. Becker.—See I.

Checker bricks for [water-gas] carburettors. Parmelee and Westman.—See VIII.

Patents.

Purification, cleansing or washing of carbonaceous and other materials. B. Laing and H. Nielsen (E.P. 250,291, 27.11.24).—The carbonaceous material, containing a loose admixture of stones or other impurities, is subjected to a preliminary heat treatment (*e.g.*, in a retort at 250—350°), the volatile products being recovered. The action of the heat is to render the carbonaceous material more porous, so that its subsequent separation from the impurities by any of the usual wet or dry methods is facilitated. W. T. K. Braunholtz.

Briquetting wood particles. H. H. Holdaway, Assr. to L. F. Pike and A. B. Flickinger (U.S.P. 1,580,299, 13.4.26. Appl., 21.1.25).—Comminuted wood material is dried and rapidly compressed, under the influence of heat, to about one-sixth of its free volume. The material may be exposed, during the process, to the action of a gaseous mixture of phenol (1 pt.), formaldehyde (3 pts.), alcohol (48 pts.), and water (64 pts.). W. T. K. Braunholtz.

Combustion, gasification, or degasification of pulverulent fuels. G. Szikla (E.P. 245,461, 4.2.25. Conv., 4.2.24).—The fuel is held in suspension in a vertical shaft, flaring upwards, by an upward current of hot air (for combustion or gasification), or hot gas (for distillation or degasification) and any particles of fuel falling from the shaft are received by a grate beneath the shaft. The grate is mounted in a chamber connected to, and of greater cross-section than, the lower end of the shaft, and the whole or a part of the air or gas is supplied through this chamber. The air is preheated by passing it through or over the fuel burning on the grate, and devices are provided within the chamber for regulating the proportion of the air passing through the grate. H. Holmes.

Heating of coke ovens. N. V. Silica en Ovenbouw Mij, Assees. of C. Otto u. Co. (E.P. 247,205, 4.2.26. Conv., 4.2.25).—The necessity of varying the height above the oven sole of the point of initiation of combustion in the flues may be due to the bottom of the charge being more rapidly coked than the top, or to the use of a richer or leaner gas, and is met by placing in the bottom of the combustion chamber a pipe-shaped brick arranged to take interchangeable pipe-shaped bricks of different lengths, giving the effect of burners of different heights. The changes are made through the roof of the combustion chamber. R. A. A. Taylor.

Heat treatment of materials for distilling, drying, or carbonising them. Thermal, Industrial and Chemical (T.I.C.) Research Co., Ltd., and D. Rider (E.P. 251,724, 27.2.25).—The material, *e.g.*, wood waste to be carbonised, is conveyed by a screw with a shallow thread of small uniform pitch through a cylindrical retort mounted in a furnace with its axis vertical. As little clearance as possible is left between the thread and the wall of the retort to enable the heating to be effected under conditions approximating to those for film distillation. The shaft of the screw is mounted to allow for expansion and contraction. The retort is preferably provided with a jacket containing molten metal to ensure a uniform temperature. The volatile products from the material escape through perforations in the wall of the screw, and are withdrawn by suction to prevent overheating or cracking. H. Holmes.

Generation of gas. E. S. Suffern (E.P. [A] 252,045, 12.1.25, and [B] 252,222, 23.1.25).—(A) The generator consists of a cylindrical or rectangular chamber lined with firebrick. Pulverised fuel and air preheated if necessary are admitted through a nozzle in a direction axial to the chamber and ignited. When a suitable temperature has been reached, the supplies of fuel and air are regulated to give a suitable gas. The stream of reacting substances impinges on the further ends of the side walls and on the opposite end wall and returns to pass out through an opening in the side near the inlet end, in its course raising the temperature of the incoming stream. The velocity of the gases is sufficient to maintain the residue from combustion in suspension, so that it passes out with the gases and is removed subsequently. The side walls of the generator may be fitted with baffles to give an eddying motion to the gas. Steam may be introduced and the gases may be otherwise enriched. (B) An oil fuel is projected axially by an air blast through an atomiser into a cylindrical or rectangular generating chamber. The heat of the partial combustion is generally sufficient to maintain the reaction, but preheating may be necessary. The gases impinge on the far ends of the side walls and on the opposite end wall of the chamber and return to an exit near the inlet end. They thus set up an eddying motion and influence thermally the reacting mixture. A trap may be introduced for collecting molten slag or solid residues of combustion. R. A. A. Taylor.

Purification of coal gas. T. V. Miles, G. W. Allott, and Newton Chambers & Co., Ltd. (E.P. 251,755, 17.4.25).—Where oxide dry purifiers are used, the gases are raised to a temperature requisite for the reaction by passing through a heat exchanger. This has for its hot element the system of leads from the hydraulic main to the condensers and scrubbers, and for its cool element the leads from the scrubbers to the purifiers. The heat exchanger is designed so that the gases follow a sinuous path, of which turns can be by-passed in order to maintain uniformity in the temperature of operation of the purifiers. R. A. A. Taylor.

Fuels for internal - combustion engines. Allgem. Ges. für Chem. Ind. (E.P. 245,072, 23.10.25. Conv., 27.12.24).—Petrol, benzol, and like fuels are enriched in respect of constituents soluble in liquid sulphur dioxide, in order to prevent "knocking," by the addition of products, boiling up to 200°, obtained by extracting mineral oil or lignite tar distillates, boiling at 150—300°, with liquid sulphur dioxide. H. Holmes.

[Alcohol-ether] fuels. A. P. Mackilligin and F. Garland (E.P. 251,157, 19.8.25).—A motor spirit consists of a mixture of alcohol and ether in proportions varying between 7 : 3 and 3 : 7 according to the compression ratio employed. To this mixture is added 0·5% of pyridine, 0·5% of caoutchoucine, and 1% of kerosene, by volume. These additions act as denaturants, and the first also as a neutralising agent. Caoutchoucine is obtained by the distillation of crude rubber at 176°. S. Bowman.

Explosive fuel mixture. Fuel mixture for internal-combustion engines. J. E. Babb, Assr. to Waverly Oil Works Co. (U.S.P. 1,579,801–3, 6.4.26. Appl., [A] 15.12.24, [B] 14.2.25, [C] 22.4.25).

—The fuel contains (A) a gum resin soluble in gasoline and an oxide of lead, (B) lead oxide and a quantity of acids obtained by distillation of rosin greater than that equivalent to the lead oxide, or (C) lead oxide and an organic acid in quantity sufficient to combine with the lead present after combustion and render it volatile. S. Bowman.

Motor fuel. L. Edeleanu (U.S.P. 1,585,473, 18.5.26. Appl., 2.12.25).—A motor fuel distilling between 130° and 220° is obtained by treating a mineral oil distillate boiling between 120° and 300° with liquid sulphur dioxide, freeing the extract from sulphur dioxide, and fractionating. W. N. Hoyte.

Cracking of hydrocarbon oils. M. Melamid (E.P. 231,190, 20.3.25. Conv., 22.3.24).—The oil to be cracked is introduced in an atomised condition, together with hydrogen, into the reaction vessel, which contains a liquid contact substance such as molten tin at 300—400° (cf. E.P. 221,559; B., 1924, 1006). The temperature is so adjusted that the oils of lower boiling point are cracked, while the fractions of higher boiling point are withdrawn unchanged from the reaction vessel, thereby avoiding coke formation. W. N. Hoyte.

Production of aromatic hydrocarbons by cracking. A. E. Dunstan, R. Pitkethly, and E. S. L. Beale (E.P. 250,701, 29.1 and 29.10.25).—The extract produced in the treatment of kerosene and heavy oils with liquid sulphur dioxide, consisting largely of higher aromatic hydrocarbons, is freed from sulphur dioxide and subjected to cracking at a temperature of 420—440° and a pressure of 400—1000 lb. per sq. in. Under these conditions an extract originally showing 1% yielded 36% boiling below 200°, the distillate consisting chiefly of benzene, toluene, and xylene. S. Bowman.

Cracking oils. Power Specialty Co. (E.P. 251,404, 24.3.25).—In a tubular cracking plant the temperature of the gases is equalised and overheating avoided by supplying excess air to the furnace, and thus producing a large volume of heating gases at a lower temperature. Two furnaces may be situated side by side, each furnace being divided by a bridge into two compartments. In the first of these combustion takes place and tubes containing oil are disposed at the sides, and also in the roof over the two compartments and as a bank in the second compartment, where they are exposed to the heating gases. The gases leaving the furnace pass to a preheater, in which the air for combustion is heated, say, to 205°. The result is that the maximum furnace temperature is reduced, say, from 1370° to 1090°, with an increase of overall thermal efficiency, say, from 58·5% to 60·2%. H. Moore.

Apparatus for cracking hydrocarbons. F. E. Wellman, Assr. to Kansas City Gasoline Co. (U.S.P. 1,581,895, 20.4.26. Appl., 21.4.20. Renewed 12.2.26).—The unit consists of two sloping tubes, connected together at one end. At the opposite end, the upper tube is connected to the oil preheater, and the lower one provided with a blow-out for the carbon, both tubes being of sufficient diameter to allow the carbon formed to circulate without blocking. The tubes are filled to about one-half of their capacity and heated to a pressure of 300 lb., when half the contents are removed as vapour, the remainder, containing particles of carbon in suspension, being blown out. The whole system is controlled by a pump the capacity of which is greater than that of the tubes. E. S. Kreis.

Method of heating [cracking] oil. F. E. Wellman, Assr. to Kansas City Gasoline Co. (U.S.P. 1,581,896, 20.4.26. Appl., 30.1.22. Renewed 12.2.26).—The vertical still is heated around its sides by means of a rotating burner, which leaves the bottom portion, where the carbon settles, unheated. This bottom portion is, furthermore, protected by a shield. E. S. Kreis.

Apparatus for hydrocarbon-oil-cracking operations. K. V. King, Assr. to Standard Oil Co. (U.S.P. 1,582,407, 27.4.26. Appl., 6.5.25).—The retort or still is electro-plated internally with chromium in order to make it resistant to corrosive action during cracking operations. S. Bowman.

Pressure oil still. R. E. Humphreys, Assr. to Standard Oil Co. (U.S.P. 1,582,929, 4.5.26. Appl., 7.1.21).—The still consists of an upper and a lower drum arranged outside a heating chamber through which tubes connecting the drums pass. The oil is fed into the heating drums from two storage drums placed above and also outside the heating chamber. S. Bowman.

Apparatus for decomposition and recombination of hydrocarbons. A. T. Stuart and G. N. Middleton, Assrs. to F. G. Clark (U.S.P. 1,584,048, 11.5.26. Appl., 5.7.17. Renewed 10.8.25).—A retort heated electrically to a cracking temperature is provided with an adjustable feed for oil and gas which is also connected to a reflux condenser fitted over the offtake of the retort. S. Bowman.

Continuously distilling and cracking hydrocarbon oils. J. W. Coast, jun., Assr. to Doherty Research Co. (U.S.P. 1,585,233, 18.5.26. Appl., 14.7.19).—A body of oil is subjected to heat and pressure and the lighter fractions are distilled off, while at the same time the products of intermediate boiling point are cracked. A portion of the oil is withdrawn and its pressure released to cause the vaporisation of the products of intermediate boiling point and these are passed into contact with the oil feed to distil off its lighter hydrocarbons. H. Moore.

Process of catalysing [hydrocarbon] oils. G. L. Pritchard and H. Henderson, Assrs. to Gulf Refining Co. (U.S.P. 1,585,263, 18.5.26. Appl., 28.2.22).—A mixture of the oil with aluminium chloride as catalyst is passed through a heating zone, and then allowed to expand freely, and the vapours of volatile hydrocarbons thus evolved are separated. W. N. Hoyte.

Conversion of [hydrocarbon] oils. J. H. Adams, Assr. to Texas Co. (U.S.P. 1,585,355, 18.5.26. Appl., 28.6.23).—Hydrocarbon oils of high boiling point are continuously converted into products of lower boiling point by heating a stream of the oil under pressure to the point of incipient vaporisation while maintaining it in liquid form. The heated oil is forced still under pressure through a cracking coil of small cross-section at a cracking temperature. In this the oil vaporises and is partially cracked, and is then expanded into a cracking chamber at substantially the same temperature and pressure, where further cracking takes place. The cracked and uncracked oils are removed from the chamber, and the vapours are condensed under pressure.

H. Moore.

Controlling the supply of steam and oil to an oil-cracking still. C. J. Greenstreet, Assr. to Gasoline Corp. (U.S.P. 1,585,381, 18.5.26. Appl., 27.12.19).—The intake pipe to the cracking still is branched; the steam branch is fitted with a shut-off valve, a regulating valve, and a valve-controlled vent to the atmosphere located between the other two valves. The oil branch is fitted with a shut-off valve.

W. N. Hoyte.

Process for making gasoline. R. C. Holmes, F. T. Manley, and O. Behimer, Assrs. to Texas Co. (U.S.P. 1,585,496, 18.5.26. Appl., 13.8.25).—Hydrocarbon oil is forced through a heated coil and drum, the vapours developing pressure till decomposition of the hydrocarbon begins. The heat applied is then reduced, whilst the drum is secured against substantial loss by radiation and vaporisation. The necessary heat is continuously supplied to the drum by the stream of oil entering from the coil, which mingles with the hot condensate from the vapours.

H. Moore.

Electrochemical treatment of unsaturated hydrocarbon compounds resulting from the cracking of oils. D. L. Thomas (U.S.P. 1,585,573, 18.5.26. Appl., 9.2.25).—The compounds are subjected to a silent high-tension electric discharge, under high pressure and at high temperature, to produce by combination of their molecules (polymerisation) a stable product of different gravity and boiling point from the compounds treated.

H. Moore.

Apparatus for treating hydrocarbon oils. O. Behimer, Assr. to Texas Co. (U.S.P. 1,582,893, 4.5.26. Appl., 15.2.24).—An internally heated still is provided with a hinged jacket formed of heat-insulating material.

S. Bowman.

Distillation [of hydrocarbon oils]. W. E. Chenot (U.S.P. 1,584,730, 18.5.26. Appl., 22.4.24).—The vapours from the still are passed up a dephlegmator, and the reflux from the dephlegmator is redistilled by heat interchange with the contents of the main still; the vapours from the redistillation are dephlegmated and condensed in the main condenser.

W. N. Hoyte.

Distilling [hydrocarbon] oils. C. M. Alexander (U.S.P. 1,585,224, 18.5.26. Appl., 9.10.19).—The oil is passed through a pipe still, into which steam is injected at several points. Oil and steam are maintained in intimate contact; light and heavy fractions are then separated by fractional condensation.

W. H. Hoyte.

Vacuum distillation of lubricating oils and the like. V. L. Oil Processes, Ltd., O. D. Lucas, and E. L. Lomax (E.P. 251,363, 4.2.25).—For the production of lubricating oils heavy hydrocarbon oils are heated in a pipe still to a temperature slightly above the b.p. of the fractions it is desired to remove, under sufficient pressure to prevent the formation of vapour. The heated oil is passed to one or more dephlegmators maintained at a reduced pressure, where the heavier oils condense. The heat of the vapours serves to drive off lighter constituents from the condensate, and a jet of superheated steam assists to carry the vapours through the column, the steam passing over with the lighter oils. The dephlegmator is maintained at a high vacuum, and condensed oil is removed by pumps from the bottom of the column. The vapours are condensed by water condensers under vacuum. Tarry matter may be removed in the first column of a series, and lighter oils in the succeeding columns. The exits from the condensers and the flow of returned oils are controlled by siphonic barometric columns. Pitch may be collected in a vacuum tank while still hot.

H. Moore.

Purification of petroleum, benzine and benzol hydrocarbons, their homologues, etc. Ges. für Wärmetechnik, and A. Ufer (E.P. 251,117, 21.5.25).—Treatment of petroleum spirit etc. with sulphuric acid is followed by the addition of water or a salt solution before the waste acid is run off. The advantages gained thereby are a more complete separation of the acid from the refined product and also the separation of resinous substances from the acid. If ammonium sulphate solution be used, the acid liquor can be returned to an ammonia saturator and the separated resins mixed with crude tar for distillation. An economy in neutralising reagents is effected by the more complete separation of the acid.

S. Bowman.

Refining [hydrocarbon] oils. R. Wiles, Assr. to Standard Oil Co. (U.S.P. 1,579,799, 6.4.26. Appl., 11.7.21).—Gases containing gasoline constituents are passed through an absorber containing reduced crude oil which is then mixed with ordinary crude oil and redistilled. The liquid constituents of the gas are thus recovered.

S. Bowman.

Process for making a highly-active charcoal in grains. H. Müller-Clemm and E. Schmidt, Assrs. to Ges. für Chem. Produktion (U.S.P. 1,586,106, 25.5.26. Appl., 24.10.24).—See E.P. 231,466; B., 1925, 904.

Manufacture of gas. M. W. Travers and F. W. Clark, Assrs. to Regenerative Coal Gasification

System, Ltd. (U.S.P. 1,587,564, 8.6.26. Appl., 12.6.24. Conv., 29.6.23).—See E.P. 210,356; B., 1924, 244.

Manufacture of gas. G. H. Kaemmerling and H. W. Benner, Assrs. to Fuller-Lehigh Co. (U.S.P. 1,587,588, 8.6.26. Appl., 2.11.20).—See E.P. 171,079; B., 1922, 974 a.

Apparatus for coking coal. R. B. Kernohan (U.S.P. 1,587,589, 8.6.26. Appl., 12.8.22).—See E.P. 202,327; B., 1924, 458.

Treatment of gas liquor and other waste liquors for the separation and recovery of phenol therefrom. LeR. W. Heffner and W. Tiddy (E.P. 244,774, 15.12.25. Conv., 16.12.24).—See U.S.P. 1,566,795-6; B., 1926, 147.

Removal of naphthalene and analogous hydrocarbons from fuel gases. H. Wade. From Koppers Co. (E.P. 252,497, 25.3.25).—See U.S.P. 1,578,687; B., 1926, 430.

Treatment of tar water. J. Y. Johnson. From Badische Anilin- & Soda-Fabrik (E.P. 252,982, 5.11.25).—See U.S.P. 1,579,957; B., 1926, 478.

Coal pulverising machines. A. Herbert, P. V. Vernon, and R. Jackson (E.P. 252,527, 2.5.25).

Method and apparatus for controlling combustion. A. E. White. From Surface Combustion Co. (E.P. 252,450, 25.2.25).

Apparatus for the combustion of elastic and/or liquid fuels. A. L. Mond. From E. H. Peabody (E.P. 252,868, 12.5.25).

Apparatus for the combustion of gaseous, pulverised or similar fuels. A. L. Mond. From E. H. Peabody (E.P. 252,869, 12.5.25).

Construction of [closures for] coke ovens. A. Beckers (E.P. 248,331, 9.12.25. Conv., 25.2.25).

Apparatus for treating [quenching] coke when discharged from retorts and the like. Drakes, Ltd., and J. W. Drake (E.P. 253,349, 29.5.25).

Generation and distribution of gas. Humphreys & Glasgow, Ltd., Assees. of J. A. Perry (E.P. 246,459, 7.1.26. Conv., 20.1.25).

Mechanically-clinkered gas producers. Humphreys & Glasgow, Ltd., and A. G. Glasgow (E.P. 253,326, 6.5.25).

Bitumen emulsions (E.P. 252,258 and 252,260).—See IX.

Purification of effluents containing phenols (G.P. 426,422).—See XXIII.

III.—TAR AND TAR PRODUCTS.

Phenolic constituents of producer-gas tar from Bohemian lignite. W. Steinkopf and T. Höpner (J. pr. Chem., 1926, [ii], **113**, 137—158).—The phenols were extracted, purified, and fractionated. The fractions of b.p. 180—190° contain phenol and *o*-cresol, those of b.p. 190—205° contain *o*-, *m*-, and *p*-cresol, whilst the fractions of higher boiling point contain phenols with longer side-chains, the xylenols, and β-naphthol. The phenolic products were identified chiefly by conversion into phenylurethanes and aryloxyacetic acids. The melting points of a series of the phenolic derivatives, and in the case of the phenylurethanes the solubilities in various organic solvents are given. The following new compounds were prepared: *phenylurethanes* of 1:2:3-xylenol, m.p. 176°, of 1:2:4-xylenol, m.p. 120°, of 1:3:2-xylenol, m.p. 133°, of 1:3:4-xylenol, m.p. 112°, of *m*-ethylphenol, m.p. 138·8°, and of *p*-ethylphenol, m.p. 120°; o-, m,- and p-*ethylphenoxyacetic acids*, m.p. 140—141°, 75—75·5°, and 96—97° respectively; 1:2:3- and 1:3:2-*xylenoxyacetic acids*, m.p. 187° and 139·5° respectively; *mesitoxyacetic acid*, m.p. 131·5°, and *pseudocumenoxyacetic acid*, m.p. 132°. J. S. H. Davies.

Constituents of bituminous tar oils rich in sulphur [ichthyol oils]. IV. H. Scheibler and F. Rettig (Ber., 1926, **59**, 1198—1202).—The presence of 2-*n*-butylthiophen in an oil from Achensee and of 3-*n*-propyl- and 2-*iso*propyl-thiophen in a similar product from Seefeld is established by treating the individual fractions of the oils with a deficiency of aluminium chloride and acetyl chloride and identifying the acetyl compounds of the thiophen derivatives thus produced as their *p*-nitrophenylhydrazones. H. Wren.

Low-temperature tar oils. G. P. Lewis (Fuel Econ., 1926, **1**, 365—367).—Low-temperature tar oils have no economic value except as fuel. When certain low-temperature tars including pitch were heated to 500° under pressure the products showed increased and decreased contents of fractions of low and high boiling point respectively. It frequently happened that other tars did not benefit by the same treatment but similar results could be produced even in those cases by the addition of certain catalysts. S. Pexton.

Determination of phenol in crude cresol. W. Qvist (Z. anal. Chem., 1926, **68**, 257—273).—In the determination of *m*-cresol in crude cresol by the method previously described (B., 1925, 164) the bulk of the phenol is found as picric acid in the precipitate of trinitro-*m*-cresol and its weight is obtained by difference after determining the trinitro-*m*-cresol as described. The filtrate from the mixed nitro-products is diluted to 230 c.c. and distilled in steam until 250 c.c. of distillate have collected. The residual solution in the distilling flask is diluted to 250 c.c. and extracted twice with 50 c.c. of toluene. The toluene solution is dried by filtration through thick filter paper and 50 c.c. are shaken with an excess of 0·1*N*-sodium hydroxide solution. The sodium picrate solution is separated and the toluene washed twice with 100 c.c. of water. Finally the excess of alkali in the aqueous layer is determined by titration with 0·1*N*-hydrochloric acid using methyl-red as

indicator, and the picric acid content of the original filtrate is calculated from the alkali consumed. Tests of the method with 10 g. of pure phenol gave an average yield of 20·6 g. of picric acid, of which the filtrate contained 2·0—2·2 g. of the acid; therefore the total picric acid found in the analysis divided by 2·06 gives the phenol content of the cresol.

A. R. Powell.

Patents.

Production of aromatic hydrocarbons (E.P. 250,701).—See II.

Purification of benzol hydrocarbons (E.P. 251,117).—See II.

Purification of effluents containing phenols (G.P. 426,422).—See XXIII.

IV.—DYESTUFFS AND INTERMEDIATES.

Sulphurisation and oxidation products of methylanthraquinone. A. Schaarschmidt and H. Lewyeff (J. pr. Chem., 1926 [ii], 113, 48).—Analysis of Cibanone Orange R (G.P. 209,232), recrystallised from much nitrobenzene, shows the presence of one atom of sulphur to each mol. of β-methylanthraquinone. An attempt to obtain an oxidation product similar to the dye by the action of persulphate on β-methylanthraquinone in sulphuric acid gave a dimorphous *substance*, $C_{15}H_8O_2$, m.p. 172° and 164°, insoluble in alkali, and easily brominated.

C. Hollins.

Behaviour of hydrated chromic oxide towards dyestuffs. H. M. Burns and J. K. Wood (J. Soc. Dyers and Col., 1926, 42, 187—190).—Hydrated chromic oxide precipitated with excess of ammonia (even after prolonged washing) retains traces of ammonia which decrease the absorptive power of the oxide for acid dyes (Ponceau R, Naphthol Yellow S, Orange II, Fast Red A, and Methyl Orange). The curves obtained are more nearly linear than those obtained with oxides containing a considerable amount of chloride. The latter showed the largest absorptive power and gave curves approaching in character an ordinary adsorption isotherm. In one case the amount of Ponceau R and Fast Red A taken up by the oxide was independent of the concentration of the solution, indicating the formation of definite compounds between the dye and the oxide. The influence of impurities on the dyeing properties of hydrated chromic oxide is given the electrical interpretation already given in the cases of titanic acid, stannic acid, and alumina (B., 1923, 544 A; 1925, 203).

R. Brightman.

Use of *m*-chlorophenol for the preparation of intermediates and the positional influence of methylthiol-, methoxy-, and chlorine on the colours of certain azo dyestuffs. H. H. Hodgson (J. Soc. Dyers and Col., 1926, 42, 175—179).—See B., 1925, 702; A., 1926, 515.

Patents.

[Vat] dyes and dyeing. J. Morton, J. I. M. Jones, B. Wylam, J. E. G. Harris, and Morton Sundour Fabrics, Ltd. (E.P. 251,491, 1.11.24).—Water-soluble derivatives of vat dyes are obtained by treating anthraquinone or indigoid vat dyes suspended in a tertiary base, such as pyridine or dimethylaniline, in the presence of a metal such as copper or zinc, with chlorosulphonic acid or its salts, fuming sulphuric acid, sulphuric anhydride, or salts of pyrosulphuric acid, small quantities of acids or salts being added to assist the reaction; the resulting products are dyestuffs and are extracted from the reaction mixture by means of a limited quantity of water. For example, a mixture containing 13·6 pts. of chlorosulphonic acid, 30 pts. of dry light pyridine, 5 pts. of Caledon Yellow G (flavanthrone), and 3 pts. of finely-divided copper is heated at 40° for 20 min., then cooled, and heated with 100 pts. of water containing 17·6 pts. of ammonia (*d* 0·880), and filtered. The resulting dark blue-black residue is extracted at 50° with 500 pts. of a 1% solution of caustic soda, and a water-soluble vat dye is isolated from the extract by addition of salt.

A. J. Hall.

Recovery of antimony in the manufacture of flavanthrone. W. A. Adamson and P. W. Carleton, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,582,328, 27.4.26. Appl., 29.2.24).—The filtrate from flavanthrone (prepared by digesting β-aminoanthraquinone with antimony pentachloride in nitrobenzene solution) is distilled *in vacuo* and the distillate consisting of antimony trichloride in nitrobenzene is treated below 50° with a calculated quantity of chlorine to convert all the antimony present into the pentachloride. The solution thus obtained can be used again for the preparation of flavanthrone. About 80% of the antimony and nitrobenzene used is recovered.

R. B. Clarke.

Vat [benzanthrone] dyestuffs. G. Kränzlein, H. Greune, R. Sedlmayr, and H. Vollmann, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,582,475, 27.4.26. Appl., 27.11.25).—New yellow vat *dyestuffs* are obtained by fusing a benzanthrone with an anhydride of an aromatic mono- or di-carboxylic acid in the presence of aluminium chloride. Bisbenzpyrenequinones are probably obtained from mono-acids, while phthaloyl- or naphthoyl-benzanthrones result from di-acids. The reaction product obtained from benzanthrone and phthalic anhydride is probably 2:3-(Bz)phthaloylbenzanthrone. For example 68 pts. by weight of benzanthrone, 90 pts. of phthalic anhydride, and 150 pts. of aluminium chloride are intimately mixed and heated to 140—160°. The melt is extracted with water and dilute hydrochloric acid, filtered, and the residue treated with dilute caustic soda to remove unchanged anhydride. The yellow-brown paste obtained gives a green hyposulphite vat which produces brilliant fast yellow shades on cotton.

R. B. Clarke.

Manufacture of 2:3-diaminoanthraquinone. Soc. of Chem. Ind. in Basle, Assees. of W. Moser (U.S.P. 1,586,911, 1.6.26. Appl., 13.4.23. Conv., 24.5.22).—See E.P. 203,051; B., 1923, 1063 A.

The product dyes cellulose acetate silk weak yellowish-brown tints.

Manufacture of vat-dyestuffs of the perylene series. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 253,311, 28.4.25).—See U.S.P. 1,551,849; B., 1925, 911.

Halogenising perylene. A. ZINKE and A. PONGRATZ, Assrs. to F. BENSA (U.S.P. 1,580,708, 13.4.26. Appl., 7.4.25).—See E.P. 232,266; B., 1925, 911.

Process for manufacturing amino-perylene-quinones. A. ZINKE, Assr. to F. BENSA (U.S.P. 1,586,729, 1.6.26. Appl., 15.6.23. Conv., 7.4.23).—See E.P. 199,721; B., 1924, 781.

Process for manufacturing vat [perylene] dyes. A. ZINKE and F. HANSELMAYER, Assrs. to F. BENSA (U.S.P. 1,586,730, 1.6.26. Appl., 14.2.24. Conv., 7.4.23).—See E.P. 213,889; B., 1925, 537.

Manufacture of vat colouring matters of the benzanthrone series. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABRIK (E.P. 252,903, 11.6.25).—See F.P. 598,752; B., 1926, 311.

Manufacture of polyazo-dyestuffs. A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 253,377, 20.7.25).—See G.P. 423,092; B., 1926, 312.

Azo dyestuffs. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. L. LASKA and A. ZITSCHER (U.S.P. 1,587,004, 1.6.26. Appl., 31.3.25. Conv., 7.6.24).—See E.P. 235,169; B., 1925, 666.

Azo dyestuffs. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. ZITSCHER (U.S.P. 1,587,060, 1.6.26. Appl., 30.11.23. Conv., 12.1.23).—See E.P. 218,568; B., 1924, 824.

Basic dyestuffs of the Malachite-Green series. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of R. REYHER (U.S.P. 1,588,052, 8.6.26. Appl., 2.9.25. Conv., 27.9.24).—See E.P. 251,511; B., 1926, 577.

Manufacture of 2-aminonaphthalene-1-carboxylic acid or its nucleal substitution products. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS & BRÜNING (E.P. 252,820, 12.3.25).—See G.P. 418,009; B., 1926, 355.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemical changes in sulphite-cellulose boiling. O. ROUTALA and J. SEVÓN (Ann. Acad. Sci. Fennicæ, 1926, **26**, [4]. Reprint, 30 pp.).—Analyses of the liquor and pulp have been made at various stages of technical sulphite-cellulose boils, and of laboratory boils controlled so that the temperature at each stage was the same as in a technical boil. The dissolution of incrusting substances begins during the first hour of the boil, and reaches an approximately constant velocity when a temperature of 80—100° is attained. It is therefore desirable to heat to 100° as rapidly as possible. Towards the end of the boil the rate of dissolution diminishes. The sulphurous acid and calcium contents of the wood increase independently of the temperature, within certain limits, and the end-point is always reached after the same time. Carbohydrates and lignin pass into solution together until 23·2% of the wood has been dissolved; lignin then dissolves alone. The carbohydrates dissolved during the first few hours are mostly pentoses, hexoses dissolving later; the ratio of pentoses to hexoses in the hemicellulose is 1 : 2. It is probable that lignin and carbohydrates are combined in wood as glucoside-like compounds, and that the principal reactions in the sulphite-boiling process are the hydrolysis of such compounds and the formation of soluble calcium ligninsulphonates. The compound of lignin with cellulose contains the constituents in the ratio 1 : 2·5, corresponding with 1 mol. of lignin (equivalent weight 360) to 5 glucose residues; the compounds of lignin with hemicelluloses contain 1 mol. of pentose and 2 mols of hexose for each mol. of lignin. During boiling the resin and fat are partially removed, but this is mainly due to mechanical action. A. GEAKE.

Wood pulp. III. Properties of the purified pulp. T. OZAWA (J. Soc. Chem. Ind. Japan, 1926, **29**, 78—84).—A comparison of ordinary bleached sulphite wood pulp with pulp purified with lime and sodium sulphite (J. Soc. Chem. Ind. Japan, 1925, **28**, 285). On heating at 95—100° for 22—84 hrs. or storing for 47 days in air, the pulp gradually becomes yellowish-brown, the copper number increases, and the content of α-cellulose decreases, these changes being more rapid and marked in the ordinary pulp than in the purified pulp. Viscose made from the purified pulp differs in many respects from viscose made from ordinary pulp, and resembles rather the viscose from cotton cellulose in respect of the changes during ageing and of the properties of the regenerated celluloses. The copper number of the cellulose regenerated from the viscose after ageing for different periods increases with the duration of ageing, whilst the viscosity decreases; here also the effects are more marked with ordinary pulp. These differences are due to the presence of degraded cellulose in the ordinary wood pulp.

K. KASHIMA.

PATENTS.

Conditioning of raw cotton stock. BORNE SCRYMSER Co., Assees. of R. B. SMITH (E.P. 242,593, 27.7.25. Conv., 8.11.24).—The production of static electricity during the working up of raw cotton stock is minimised by treating the stock, either in bulk or before the completion of the drawing operation, with not more than 4% and preferably not less than 0·5% (on the weight of cotton) of a non-volatile, stable oil. The most satisfactory results are obtained with a pure mineral oil having a viscosity between 25 sec. Saybolt at 21° and 300 sec. Saybolt at 100° and density between 0·8 and 0·966. The oil may be removed from the finished material by the use of suitable emulsifying agents.

D. J. NORMAN.

Treatment of silk. Brit. Silk Research Assoc., W. S. Denham, and W. Brash (E.P. 252,064, 13.2.25).—The mineral content of silk may be considerably reduced, leaving at the same time a substantially neutral product, by immersing the silk in an acid solution of such p_H (approximately 3·8) that the silk is brought to an isoelectric condition. For example, 50 lb. of degummed silk are immersed in 400 litres of water of 3° hardness. Dilute sulphuric acid is then added until a sample of the water just turns bromophenol-blue yellow (p_H slightly below 3·8). The silk is centrifuged and the process repeated three times, the material being finally washed in the centrifuge with tap water until the wash water is neutral to methyl-orange. The ash content of a sample of waste silk treated by this process was 0·71% as against 3·16% when the silk was degummed and washed in the usual way. D. J. Norman.

Production of waterproof textile material, paper, and the like. C. Knopf (E.P. 251,126, 2.6.25).—The disadvantages associated with the production of waterproof fabric or paper by the action of concentrated solutions of metal chlorides (cf. G.P. 377,659; B., 1923, 971 a) are eliminated by adopting the following procedure. The material is dried before entering the bath, and, on leaving it, is scraped on both sides, squeezed to remove superfluous chemicals, and passed over a heating drum to assist amyloid formation. The material is then immediately cooled and passed into the first of a series of washing baths, after which it is subjected to a number of sprays in such a manner that the sheet is not broken. When completely washed, it is dyed or otherwise treated, and slowly dried. D. J. Norman.

Production of artificial filaments. L. A. Levy (E.P. 251,680, 30.1.25).—A semi-dry-spinning process is used in which the volatile solvent is removed partly by evaporation and partly by a fixing bath. The filaments are spun in a closed or nearly closed chamber in which air at any desired temperature is circulated; the fixing bath, optionally heated, may be inside or outside this chamber, and by adjusting the temperature of the air and the position of the nozzle relative to the fixing bath, it is possible to control the lustre and appearance of the filaments within fairly wide limits. In general about 80% of the solvent should be removed by evaporation. D. J. Norman.

Production of alkali-cellulose. H. Hawlik and O. Sindl (E.P. 229,678, 18.2.25. Conv., 18.2.24).—The waste lye from the preparation of alkali-cellulose is used to effect a partial mercerisation and purification of succeeding batches of cellulose, until its caustic soda content falls to about 135—140 g. per litre and its hemicellulose content reaches about 60 g. per litre. The partly mercerised cellulose is in each case finally mercerised with fresh liquor. In general the cellulose should be treated three times with lye from a previous operation and twice with fresh lye. In this way a considerable economy in the consumption of caustic soda is effected and at the same time an alkali-cellulose of lower hemicellulose content is obtained. D. J. Norman.

Coating of paper, fabrics and other web and sheet material. J. D. and D. MacLaurin (E.P. 251,681, 2.2.25).—Paper and similar materials are coated with a low-grade material and subsequently, after partial drying, with a high-grade finishing material by means of spraying devices operated by means of compressed air or steam, the paper being drawn in open width beneath the spraying devices. A. J. Hall.

Manufacture of artificial silk and of artificial threads or filaments. Soc. pour la Fabr. de la Soie "Rhodiaseta" (E.P. 238,842, 24.1.25. Conv., 22.8.24).—See U.S.P. 1,583,475; B., 1926, 580.

Process for rendering nitrocellulose yarns incombustible. J. R. Lavaud (E.P. 251,227, 14.8.25. Conv., 23.4.25).—See F.P. 600,852; B., 1926, 483.

Apparatus for the recuperation of the lye from dissolved cellular substances. G. Mosebach (U.S.P. 1,587,345, 1.6.26. Appl., 5.7.24. Conv., 7.8.23).—See G.P. 398,041; B., 1925, 986.

Stencil sheet. S. Horii (U.S.P. 1,587,954, 8.6.26. Appl., 27.6.25).—See E.P. 250,798; B., 1926, 534.

Method of drying material [paper]. J. E. Alexander (E.P. 243,762, 30.11.24. Conv., 28.11.24).

Recovery of sodium hydroxide or carbonate (E.P. 252,304).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Contraction [of cellulose fibres] on mercerisation. W. Gordon (Kolloid-Z., 1926, **39**, 107—110).—Expressions are developed for the contractive force operating on cellulose fibres during mercerisation. The assumption is made that the fibres consist of cylinders of an amorphous cementing substance in which regularly oriented liquid crystallites are dispersed. By making certain assumptions the contractive force is calculated for cases where the crystallites are parallelopipeds, cylinders, ellipsoids, or cones, and in all but the last (which is held to be an improbable shape) the value obtained is of the order of magnitude of that observed. N. H. Hartshorne.

Chemistry of dyeing processes [with vat dyes]. K. Brass and M. Gayled (Rev. gén. Mat. Col., 1926, **30**, 130—132, 165—168).—Previous conclusions (B., 1925, 986) concerning the much stronger affinity of cotton for the leuco-anthraquinone vat dyestuff acids than the corresponding alkali salts are confirmed and extended to Hydron and indigoid dyes. A. J. Hall.

PATENTS.

Dyeing cellulose esters and ethers. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 243,737, 25.11.25. Conv., 25.11.24).—Slightly basic monoazo dyestuffs containing a sulphamino-group in the diazo-component are particularly suitable for dyeing cellulose esters in intense fast shades. Suitable golden-yellow, orange, and reddish-orange dyes are obtained from diazotised 4-aminobenzene-1-sulphamide and aminocresol ether, diazotised 2-nitro-1-aminobenzene-4-sulphamide and *m*-toluidine, and diazotised 4-aminobenzene-1-sulphamide and α-naphthylamine respectively. A. J. HALL.

Dyeing of cellulose ester artificial silks. C. M. BARNARD, and BRIT. ALIZARINE CO., LTD. (E.P. 252,240, 15.11.24).—Cellulose esters are dyed with substances having the general formula $A{\cdot}N(Y{\cdot}CO_2H)R^1$ or $A{\cdot}N(Y{\cdot}CO_2H)Z{\cdot}CO_2H$ in which A is a non-sulphonated aryl dye nucleus, N is an atom of nitrogen, R^1 is an atom of hydrogen or an alkyl-group or other substituent, and Y and Z are the same or different aliphatic chains which may be branched or straight, substituted or unsubstituted. The non-sulphonated aryl dye nucleus may be any of the well-known dye groupings such as anthraquinone or its derivatives, azo compounds, indigo or its derivatives, but it is preferable to maintain the molecular weight as low as possible. One group of especially suitable compounds of this type is formed by the condensation of 1 mol. of an amino- or imino-derivative of a non-sulphonated aryl dye nucleus with at least 1 mol. of an aliphatic polycarboxylic acid. Two general methods for preparing suitable substituted glycine dyes are (1) treatment of 1 mol. of an aromatic amine with 1 mol. of an aldehyde, 1 mol. of sodium bisulphite, and 1 mol. of potassium cyanide, and subsequent hydrolysis with boiling caustic soda or sulphuric acid of the nitrile thereby formed, and (2) condensation of an aromatic amine with a halogenated aliphatic acid other than acetic acid. A. J. HALL.

Dyeing of cellulose ester artificial silks. BRIT. ALIZARINE CO., LTD., and C. M. BARNARD (E.P. 252,646, 15.11.24 and 4.7.25).—Azo and anthraquinone compounds having the general formula A·X·Y·COOH, in which A is a non-sulphonated aryl dye nucleus, X is an atom of oxygen or sulphur, and Y is a straight or branched substituted or unsubstituted aliphatic chain, are suitable for dyeing cellulose esters. It is desirable that A has a low molecular weight. One group of such dyes consists of derivatives of glycollic or thioglycollic acid or homologues of these acids in which a hydrogen atom attached to the oxygen or sulphur atom is replaced by a non-sulphonated aryl dye nucleus. A suitable azo dye is prepared by condensing *p*-nitrophenol with chloroacetic acid, then reducing the nitro-group to an amino-group, and afterwards diazotising and coupling with β-naphthol. A bright bluish-red anthraquinone dye is prepared by boiling 2 : 4-dibromo-1-aminoanthraquinone with sodium sulphide, and condensing the product with chloroacetic acid. A. J. HALL.

Dyeing cellulose esters and ethers. C. E. MÜLLER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,587,669, 8.6.26. Appl., 24.1.25. Conv., 29.1.24).—See E.P. 228,557 ; B., 1925, 845.

Machines for the dyeing and like treatment of hanks. P. CALDWELL, and BRIT. COTTON & WOOL DYERS' ASSOC., LTD. (E.P. 252,507, 6.4.25).

Vat dyes and dyeing (E.P. 251,491).—See IV.

Coating fabrics etc. (E.P. 251,681).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Mechanism of the pyrites roasting process. G. F. HÜTTIG and P. LÜRMANN (Z. angew. Chem., 1926, 39, 759—765).—By heating iron pyrites and ferric oxide separately in atmospheres containing mixtures of oxygen, sulphur dioxide, and sulphur trioxide at 700° and analysing the gaseous and solid products, it is shown that the reaction between pyrites and oxygen is reversible and that the final product in all cases is a homogeneous solid solution of oxide and sulphide; on cooling, these interact with the formation of ferrous sulphate and oxide, thus: $4Fe_2O_3+FeS=FeSO_4+8FeO$. The composition of the solid solution is given by the equation $1/n=13{\cdot}0-6{\cdot}66 \log (p_{SO_2}/p_{O_2})$, where $1/n$ is the Fe:S atomic ratio. These facts explain the presence of 3—6% of residual sulphur in all samples of commercial burnt pyrites. A. R. POWELL.

Interaction of oxides of nitrogen with arsenious acid and with sulphurous acid in presence of sulphuric acid of varied strength. T. L. BAILEY (62nd. Ann. Rept. on Alkali etc. Works, 1926, 12—25).—Experiments were performed in order to establish the conditions necessary for the successful replacement of sulphuric acid by water in the final washing of the exit gases from the chamber sulphuric acid process. (1) Sodium nitrite solution was added to an excess of sulphuric acid of varying strength, a measured volume of air passed through, and the residual nitrite titrated. The loss increased with the strength of the acid from *d* 1·0 to *d* 1·47, and then decreased rapidly. The preliminary increase shows that nitrososulphuric acid cannot produce nitrous acid alone on decomposition with water but a mixture of oxides of nitrogen. (2) The reaction of nitrous acid with arsenious acid was found to be extremely slow in presence of sulphuric acid. A maximum is again found between *d* 1·39 and *d* 1·47. (3) Excess of sulphurous acid in presence of water causes evolution of nitrous oxide from nitrous acid with production of hydroxyamidosulphuric acid $HONH(SO_3H)$. (4) Excess of nitrous acid interacts with sulphurous acid in presence of water with evolution of nitrous oxide. If the water contains sulphuric acid the nitrous oxide is progressively replaced by nitric oxide, no nitrous oxide being formed with a solution of *d* 1·225 or above. The experiments summarised under (3) and (4)

were carried out with exclusion of oxygen and at air temperature. The rate of reduction to nitrous oxide is slow in case (4). Nitric oxide is similarly slowly reduced to nitrous oxide by sulphurous acid in presence of sulphuric acid of greater dilution than d 1·07. Nitrous oxide may well be produced in this way in the early chambers in the process in the neighbourhood of steam jets or water sprays. These reactions harmonise well with Divers' theory of the chamber process (B., 1911, 10, 594).

C. IRWIN.

Determination of the acidity of ammonium sulphate. H. M. LOWE (Gas World, 1926, 94, Coking Sec., 66—68; cf. B., 1926, 358).—Two solutions of ammonium sulphate were made up, from the commercial and the pure salt respectively. Equal quantities of methyl-red were added to each and the commercial solution was brought to the same state of neutrality as the pure solution with 0·1N-caustic soda. To portions of the commercial solution 0·1N-sulphuric acid was added to give solutions containing 0·01 and 0·02% of free acid and the colours of these solutions were matched by adding 0·1N-sulphuric acid to portions of the pure solution. Much less acid was required in the case of the pure solution, indicating the presence of some "buffer," probably pyridine, in the commercial solution. It is recommended that each works should determine periodically the relation between the acidity of its salt determined by potentiometric and colorimetric methods. Electrometric titration methods offer little advantage over colorimetric titration methods, the only trustworthy method being to determine the electromotive force of a solution against a standard cell potentiometrically.

S. PEXTON.

Rapid method for the analysis of hydrosulphide liquors. K. PAULI (Z. anal. Chem., 1926, 68, 286—299).—Commercial hydrosulphide solutions may contain as impurities sulphide, thiosulphate, sulphite, carbonate, and bicarbonate. Analysis of these solutions by Wöber's method (B., 1920, 625 A) gives erroneous results owing to the difficulty of completely precipitating the sulphide with zinc carbonate. The following modification based on the same general principles overcomes this difficulty. Two series of tests are made one on the solution (A) as received and the other on the same amount of solution after previous treatment with a definite amount of barium hydroxide (B) and decantation of an aliquot part. The iodine consumption, a and b c.c. respectively, is ascertained by adding an excess of 0·1N-iodine and titrating with 0·1N-thiosulphate. In the same solutions the amount of hydriodic acid formed in the first titration is determined by adding potassium iodate, again titrating with thiosulphate and deducting the c.c. of thiosulphate first used giving c and d c.c. of thiosulphate corresponding with the "iodine acidity." Two further aliquot parts of (A) and (B) are measured into an excess of saturated mercuric chloride solution, 5 c.c. of saturated ammonium chloride solution and a few drops of methyl-orange are added and the acidity determined by titration with 0·1N-sodium hydroxide (e and f c.c.). Finally a further two aliquot parts of (A) and (B) are titrated directly with 0·1N hydrochloric acid allowance being made in (B) for the barium hydroxide added; alkalinity $=g$ and h c.c. of 0·1N-acid. Then $(a—b)$=sulphite, $(f—a)/2$ =thiosulphate, $(2b—f—3d)/2$=sulphide, $(2b—f+d)/2$=hydrosulphide, $(3d—2b+f)/4$=bicarbonate, and $(2b—3d—2e+f)/2$=carbonate. The results obtained for g ($=\frac{1}{2}$ hydrosulphide+sulphide+$\frac{1}{2}$ sulphite+carbonate) and for h ($=\frac{1}{2}$ hydrosulphide+sulphide) serve simply as checks on the other results. If the value for sulphide is negative bicarbonate is present, if positive, bicarbonate is absent. If free alkali is present, d represents its amount and bicarbonate and hydrosulphide are absent.

A. R. POWELL.

Preparation of pure barium salts. D. RAQUET (Ann. Chim. Analyt., 1926, 8, 161).—Owing to their comparatively low solubility most of the salts of barium are prepared in a pure state by recrystallisation. The chloride may be purified by precipitation as chromate by an acetic acid solution of ammonium dichromate at boiling point. The precipitate of barium chromate is dissolved in dilute hydrochloric acid, hydrogen sulphide passed through, and chromium hydroxide separated by boiling with pure barium carbonate; or, the hydrochloric acid solution of the chromate may be treated with barium peroxide, when the hydrogen peroxide formed reduces the chromic acid and the excess of barium peroxide precipitates the chromium hydroxide. In the filtrate the barium will be present as pure chloride. Other salts are obtained by precipitating the carbonate with ammonium carbonate and dissolving in the appropriate acid.

D. G. HEWER.

Preparation of calcium salts free from barium and strontium. D. RAQUET (Ann. Chim. Analyt., 1926, 8, 161—162).—A 10% solution of the salt to be purified is shaken with calcium sulphate which precipitates the greater part of the barium and strontium. To the filtrate is added a one-tenth of its volume of a 20% solution of ammonium chromate and 1 vol. of 60% alcohol. After keeping for some time the chromates of barium and strontium are filtered off, the liquid is distilled to recover the alcohol, and then poured, little by little, into a cold solution of sodium carbonate, whereby a white precipitate of pure calcium carbonate is formed.

D. G. HEWER.

Preparation of salts of strontium free from barium and calcium. D. RAQUET (Ann. Chim. Analyt., 1926, 8, 162).—To a 10% solution of the strontium chloride to be purified a tenth of its volume of a 3% solution of ammonium chromate is added and after shaking and filtering if necessary, the mixture is left for 24 hrs. To the solution, thus freed from barium, a third of its volume of 20% ammonium chromate is added, and after shaking and filtering the precipitate of strontium chromate is washed by decantation with 60% alcohol, and decomposed cold with a 1% solution of sodium carbonate.

D. G. HEWER.

Utilisation of seaweed and marine plants. M. DESCHIENS (Chim. et Ind., 1926, **15**, 675—698).—Descriptions are given of the manufacture from seaweed of iodine and potassium salts, alginates, and agar-agar. The carbonisation of seaweed with production of a fuel gas and ashes from which iodine and potash are recovered, has not yet proved a commercial success, owing to the loss of iodine during the process. B. W. CLARKE.

Determination of iodine in natural waters. H. W. BRUBAKER, H. S. VAN BLARCOM, and N. H. WALKER (J. Amer. Chem. Soc., 1926, **48**, 1502—1504).—1—2 litres of water, evaporated to 200 c.c., are boiled with standard sodium hypochlorite solution after addition of 40—60 c.c. of phosphoric acid solution made by diluting 85% acid with an equal volume of water. The iodine is converted into iodate. Potassium iodide solution is added and the liberated iodine, six times as much as that present in the sample, titrated with sodium thiosulphate (cf. Hunter, J. Biol. Chem., 1909, **7**, 336). The use of sulphuric acid in place of phosphoric acid gives uncertain results. The iron present must be less than 20 pts. per million. When bromides are present longer boiling is necessary. S. K. TWEEDY.

Action of nitric acid on aluminium. UCHIDA and SASAKI.—See X.

PATENTS.

Manufacture of chemically pure hydrochloric acid. VER. FÜR CHEM. U. METTALLURGISCHE PRODUKTION (Addn. 30,237, 9.3.25, to F.P. 564,963, cf. E.P. 196,258; B., 1924, 378 A).—Hydrochloric acid formed by the action of chlorine on organic compounds is purified by passing over mineral gels, *e.g.*, of silica, the rare earths, titanium and zirconium oxides. E. S. KREIS.

Recovery of sodium hydrate [hydroxide] or carbonate [from alkali lyes from treatment of cellulosic materials]. L. N. TAYLOR (E.P. 252,304, 28.12.25).—Residual liquor obtained from the treatment of cellulose material with caustic soda, is treated with magnesium bicarbonate solution, heated to 85°, allowed to settle, the clear liquid siphoned off, and the precipitate passed through a filter. The liquid is further heated to 100° to decompose the sodium bicarbonate and then causticised in the usual manner. The non-cellulose organic compounds are recovered as insoluble organic magnesium compounds which may be incinerated for fuel or distilled for production of acetic acid, acetone, etc. H. ROYAL-DAWSON.

Process of forming sodium compounds. C. SUNDSTROM and G. N. TERZIEV, Assrs. to SOLVAY PROCESS Co. (U.S.P. 1,583,662, 4.5.26. Appl., 8.11.23).—The compound $Na_2CO_3,3NaHCO_3$ as prepared by the process described in E.P. 202,678 (B., 1923, 1020 A) contains sodium carbonate as an impurity. A pure product may be obtained by heating dry refined sodium bicarbonate in a loosely-covered, shallow dish for about 8 hrs. at 120° in a current of steam at a pressure of 13—14 lb. Large needle-shaped crystals are thus obtained. R. B. CLARKE.

Manufacture of caustic soda. A.-G. FÜR STICKSTOFFDÜNGER (G.P. 427,086, 22.1.24).—Calcium carbide is treated with sodium carbonate solution of *d* 1·1 in a pressure vessel. The temperature needed to complete the reaction is developed by the evolution of acetylene: $CaC_2+2H_2O+Na_2CO_3=2NaOH+CaCO_3+C_2H_2$. Calcium carbonate is precipitated in a form readily separated by filtration. E. S. KREIS.

Manufacture of alkali carbonates or bicarbonates and hydroxides. E. HENE (G.P. 427,087, 11.1.24).—Hydrocyanic acid is made to react with lime, and the resulting calcium cyanide treated with alkali sulphate. The resulting alkali cyanide is decomposed with carbon dioxide, to give hydrocyanic acid, which is used again, and alkali carbonate. E. S. KREIS.

Manufacture of alumina and aluminium sulphate. L. G. PATROUILLEAU, and SOC. ANON. ALUMINE ET DÉRIVÉS (Addn., 30,170, 13.3.25, to F.P. 586,684; cf. E.P. 240,435, B., 1925, 999 A).—The solution obtained by treating bauxite with sulphuric acid is reduced to convert the ferric salts into ferrous salts and then electrolysed, the precipitation of basic aluminium sulphate being induced by adding a dilute solution of carbonates or hydroxides of the alkalis or alkaline-earths. E. S. KREIS.

Removing sulphur from technical barium carbonate. RHENANIA VER. CHEM. FABRIKEN, J. MARWEDEL, and J. LOOSER (G.P. 427,223, 5.1.22).—Barium carbonate precipitated from barium sulphide solution by means of carbon dioxide in the presence of alkali carbonate, is purified by heating the reaction mixture to above 100° with alkali hydroxide with the addition of alkali carbonate if desired. On washing the barium carbonate is obtained pure. E. S. KREIS.

Production of mixtures of hydrogen and nitrogen. E. EDWIN (G.P. 427,542, 6.4.25).—In a cyclic process a carbon-containing substance, gaseous, liquid, or solid, is introduced into a high-tension arc together with steam and the resulting carbon monoxide and hydrogen, together with any of the unchanged original substances and any nitrogen from a former cycle, are lead away to a gas producer to which also equivalent proportions of steam and air are supplied. The mixture of gases from the producer, now enriched with the nitrogen from the air, is sprayed with water, a portion is led back to the arc, and the excess caused to react by passing it over a contact material, whereby as a result of the reaction between carbon monoxide and steam, hydrogen and carbon dioxide are formed, the latter being removed in the usual way. E. S. KREIS.

Production of sulphuric acid from sulphur-dioxide-containing-gases. METAL TRADERS TECHNICAL, LTD. From METALLBANK U. METALLURGISCHE GES. (E.P. 253,344, 21.5.25).—See G.P. 421,786; B., 1926, 319.

Production of phosphorus products. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of G. PISTOR and E. BORSBACH (U.S.P. 1,586,115, 25.5.26. Appl., 6.2.25).—See E.P. 229,282; B., 1925, 499.

Separation of hafnium and zirconium. D. COSTER and G. VON HEVESY, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,586,241, 25.5.26. Appl., 15.7.24).—See E.P. 219,024; B., 1925, 670.

Base-exchange silicate. H. J. WHEATON, Assr. to AMER. DOUCIL Co. (U.S.P. 1,586,764, 1.6.26. Appl., 28.3.22. Conv., 25.1.22).—See E.P. 177,746; B., 1922, 372 A.

VIII.—GLASS; CERAMICS.

Determination of iron in red lead [for glass manufacture]. J. F. SACHER (Farben-Ztg., 1926, **31**, 2131—2132).—Manufacturers of "crystal glass" demand a special quality of red lead containing not more than 0·005% and in some cases 0·004% of iron. While the need for such low percentages is queried, precautions necessary for their determination are outlined. A sytem of air filtration is required owing to the iron content of dust in works. Discrepancies having been found in analyses of the same red lead by gravimetric, volumetric, and colorimetric methods, a standard method is advisable. The most exact and rapid is the colorimetric one, using potassium thiocyanate, the iron being in sulphuric not hydrochloric acid solution. Excess of the latter acid affects the colour and attempts to evaporate off the excess may lead to the formation of insoluble basic salts. The presence of bismuth is also a disturbing factor in the analysis. S. S. WOOLF.

Mullite refractories formed by calcining cyanite; their industrial application. M. L. FREED (J. Amer. Ceram. Soc., 1926, **9**, 249—256).—Crude, Indian cyanite was converted entirely into interlocking mullite crystals and a silicious glass on firing to 1450°. Bricks made either from pure cyanite, pure mullite, various mixtures of cyanite with clay, or mullite with clay were fired to 1500° and tested for refractoriness-under-load, softening point, absorption, spalling, thermal expansion, and resistance to slag attack. The results indicated certain industrial possibilities; whether pure or bonded with clay, mullite produced refractory bricks capable of withstanding the laboratory tests for high-grade refractories. The specimens were highly resistant to deformation under load and to spalling and showed a uniform rate of thermal expansion. F. SALT.

Cyanite-clay refractories. I. H. C. HARRISON (J. Amer. Ceram. Soc., 1926, **9**, 257—271).—A study of the dissociation of cyanite into mullite under heat treatment indicated that, with fine material, dissociation begins, and is completed, at lower temperatures than with coarse material. With the latter, dissociation is rapid at 1300° but is not complete at 1350°, whereas with the former it is less abrupt but is complete below 1350°. Temperature is a more important factor than time in governing the rate of inversion. Trial bricks were made, using different clays as bonding material. With finely ground, uncalcined cyanite, 10% of clay gave sufficient dry strength. The coarser the cyanite, the more bonding clay was required. The physical properties of the bricks varied only slightly with the different bonding clays. Raw cyanite was used successfully in the making of bricks, but the firing expansion varied greatly with the grain-size of the material. Bricks made from calcined cyanite exhibited little change in volume on firing. Microscopical examination revealed little interaction between the cyanite particles, or their dissociation products, and the refractory bond. Better results were obtained by the use of finely ground cyanite, ball clay as bonding material, and magnesia in the form of talc as a flux, considerable development of mullite crystals in non-parallel orientation being observed. F. SALT.

Equilibrium considerations of cyanite-clay refractories. II. W. J. MCCAUGHEY and H. C. HARRISON (J. Amer. Ceram. Soc., 1926, **9**, 271—278).—Experiments with fired mixtures of cyanite and diaspore indicated that the amount of mullite produced was increased both by fine grinding of the materials and by the addition of magnesia. The mullite crystals form at the edges of the cyanite particles and grow in all directions into the interstitial spaces. F. SALT.

Effect of atmospheric conditions on the load test for refractories. L. F. Sheerar (J. Amer. Ceram. Soc., 1926, **9**, 279—289).—The effect of oxidising, neutral, and reducing furnace atmospheres on two refractory clays in the under-load test for refractoriness was determined. Varying percentages of iron pyrites were added to the clays. Nitrogen was used for the neutral atmosphere, and air which had previously been passed over charcoal at 600—750° for the reducing atmosphere. The results showed that in fireclays having a given iron content, the contraction at high temperatures was greater, and took place earlier, in a reducing atmosphere. In a given atmosphere, an increase in iron content caused an increase in expansion as the temperature was raised, and a contraction at lower temperatures. The literature of the subject is reviewed (cf. also Vickers and Theobald, B., 1925, 716, 849). F. SALT.

Determination of the bulk and pore volumes of refractory materials. M. L. HARTMANN, O. B. WESTMONT, and S. F. MORGAN (J. Amer. Ceram. Soc., 1926, **9**, 298—310).—The bulk volume of refractory materials was determined by a sand displacement method, and the pore volume by a gas porosimeter having a vertical U-tube manometer. The error in determining the porosity by these methods was less than 0·1%. A comparative series of tests showed that, on the whole, slightly higher figures for porosity were

obtained as compared with the water absorption test. F. SALT.

Mercury balance for measuring the bulk volume of bricks. A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1926, 9, 311—318).—The apparatus consists of a mercury trough, over which is a saddle arrangement to which weights can be applied. Whole bricks are immersed in the mercury by applying weights to a pan attached to the saddle. The bulk volume is calculated from the weight required to balance the upward thrust of the mercury and the density of the mercury. F. SALT.

Investigation of checker bricks for [water-gas] carburettors. C. W. PARMELEE and A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1926, 9, 290—297).—With the object of determining the best type of brick for use in carburettors in the manufacture of water-gas, and of developing suitable laboratory tests, a series of industrial and laboratory tests was carried out on a number of commercial bricks of different types. Data are presented for criticism, definite conclusions being reserved for a final report. F. SALT.

Laboratory muffle kiln [for testing ceramic products]. L. M. MERRITT (J. Amer. Ceram. Soc., 1926, 9, 324—325).—A muffle, having walls about 1 in. thick, is made in one piece over a form similar to a saggar form. The kiln is so constructed that the gases of combustion pass almost completely around the muffle. Hence, a uniform temperature is easily maintained within the muffle, and the difference in temperature between the outside and the inside is small. F. SALT.

Use of eutectics as glazes. H. M. KRANER (J. Amer. Ceram. Soc., 1926, 9, 319—323).—The possibilities are discussed of utilising the high-silica eutectics of ternary systems such as $MgO—Al_2O_3—SiO_2$ as glazes on ceramic bodies. Old plaster moulds were successfully used in a glaze of the eutectic type in which calcium oxide was the only flux. This glaze was compounded as follows: old moulds 46·6%, clay 24·2%, flint 29·2%. F. SALT.

PATENTS.

Manufacture of burnt building materials or the like from clay. H. GRONROOS (E.P. 238,229, 4.8.25. Conv., 6.8.24).—About 10 kg. of a mixture consisting of about 200 kg. of pulverised clay, 100 kg. of pulverised cryolite (previously annealed in a furnace at 600—700°), 10 kg. of iron oxide, and 400 kg. of water are kneaded with 1000 kg. of raw clay. The mixture is cut into blocks and burnt in a tunnel kiln at about 800° for 4 hrs. The burnt blocks are crushed and about 50 pts. by weight of the crushed material are mixed with 50 pts. by weight of clay, and the mixture is rolled, crushed, and mixed again before being moulded into bricks which are passed through a tunnel kiln and burnt at about 900°. H. ROYAL-DAWSON.

Refractory acid-resisting materials. A. WOLFSHOLZ (F.P. 602,475, 25.8.25).—Thoria or zirconia, preferably in a colloidal form, is mixed with a solution of calcium, aluminium, or magnesium phosphate in phosphoric acid and the mixture is shaped and fired at a red heat. A. R. POWELL.

Smoke consumption in round pottery kilns. INGENIEURGES. FÜR WÄRMEWIRTSCHAFT A.-G. (G.P. 424,717, 8.5.23).—Provision is made for admitting warm secondary air at a number of points behind the glost oven. The greater the carbon monoxide content and the rise in temperature of the gases of combustion, the further the air inlet is made to recede toward the chimney. Complete combustion of the carbon monoxide is attained by the time the gases reach the chimney, a sufficiently high firing temperature being maintained at the same time. F. SALT.

IX.—BUILDING MATERIALS.

High-alumina hydraulic cements. P. H. BATES (Ind. Eng. Chem., 1926, 18, 554—559).—A brief review is given of the composition and properties of aluminous cement, compared with those of Portland cement, with especial reference to its rapid-hardening properties and resistance to attack by salt and sulphate waters. B. W. CLARKE.

Asphalt emulsions. DE KADT.—See II.

PATENTS.

Preserving wood. N. A. ALEXANDERSON (E.P. 229,296, 10.2.25. Conv., 15.2.24).—Wood is impregnated with the liquid resin obtained as a by-product in alkali-cellulose manufacture, which may be mixed with peat-tar or wood-tar and is dissolved in petroleum burning oil (*d* 0·850—0·900, flash point 75—100°), to which is added siccatives, such as oleates and resinates of lead, manganese, or cobalt, and disinfecting agents, *e.g.*, cresols, xylenols, phenol ethers, etc. H. ROYAL-DAWSON.

Bitumen emulsions. H. W. HUTTON and C. W. FULTON (E.P. 252,258, 23.2 and 3.4.25).—Bituminous materials are heated in an apparatus provided with a stirrer and incorporated with a small proportion (1—5%) of a sulphonated oil or fat in the acid state before neutralisation. The mixture is then incorporated with a sufficient quantity of a hot solution of caustic soda or ammonia to neutralise the sulphonated oil, and hot water added in such proportion that the finished product contains about 50% of water. H. ROYAL-DAWSON.

Bituminous emulsions. G. S. HAY (E.P. 252,260, 24.2.25).—A proportion up to about 10% (by weight on the bitumen) of an emulsifying agent comprising a resin, resin acid, or resin oil, is added, with agitation, to molten bitumen. After thorough incorporation, a hot solution of caustic soda is added and the agitation of the mixture continued until emulsification has been effected. In a modification of the process the resin is dissolved in turpentine and the solution mixed with the molten bitumen, the alkali being omitted. H. ROYAL-DAWSON.

Wood preservation. A. M. Howald, Assr. to Grasselli Chemical Co. (U.S.P. 1,585,860, 25.5.26. Appl., 23.1.24).—See E.P. 228,119; B., 1925, 284.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Equilibrium relations between iron, oxygen, and carbon. R. Schenck (Stahl u. Eisen, 1926, **46**, 665—682).—It is established from data on indirect reduction of iron oxides by carbon monoxide, that in addition to magnetic iron oxide and ferrous oxide, solid solutions of these in one another and in iron take part in the equilibria as oxygen-containing phases. The probable presence of an iron suboxide, stable only below 720°, is deduced from these equilibria. The equilibria for the system iron, iron carbide, carbon monoxide, carbon dioxide cannot be determined experimentally owing to simultaneous oxidation, but data for the system iron, iron carbide, methane, hydrogen are given, and from these the constants for the first-named system are calculated. The results are used to deduce the conditions for indirect cementation of iron by carbon monoxide and methane as well as for direct cementation processes. From the equilibrium between iron and carbon monoxide below 600°, the existence of a percarbide of iron, with a considerably higher carbon vapour pressure than cementite or elementary carbon, is rendered probable. The equilibrium isotherms for carbon monoxide–carbon dioxide in the presence of iron are determined, and from them a space model showing the relation between temperature, pressure, and concentration in the gaseous phase is constructed; from this is derived a curve for the relation of gas composition to temperature which is very similar to curves experimentally obtained from observations on blast furnaces Information on the equilibria in ore-roasting processes is derived from these isotherms.

L. M. Clark.

Metallic cementation and the coating of ferrous alloys with aluminium. J. Cournot (Rev. Mét., 1926, **23**, 219—232; cf. B., 1926, 365).—After 5 or 6 normal cementations (4 hrs. at 900—950°) the efficiency of the powdered ferro-aluminium becomes impaired and the coatings produced become thinner and poorer in quality. The inner layer produced in cementing steel of eutectoid composition is quite regular and continuous but very thin (0·03 mm.) and the outer layer is 0·08 mm. thick. The thickness of the layers produced in grey cast iron are thinner than those in eutectoid steel and on subsequent heating in an oxidising atmosphere there is no marked increase in thickness. Coatings formed on ferrous alloys by this method of cementation seem to be limited to uses below 1050—1100°.

M. Cook.

Cementation of ferrous alloys by tungsten. J. Laissus (Rev. Mét., 1926, **23**, 233—242; cf. B., 1926, 278).—The bright outer layer on steel cemented with ferrotungsten contains tungsten carbide, and is formed at a lower temperature (800°) than is the corresponding layer containing chromium carbide when cementing with ferrochrome, which necessitates a temperature of 1100°. The layer produced on a steel by cementing with ferrotungsten for 10 hrs. at 1100° is destroyed by scaling after exposure for 5 hrs. at 950° in an oxidising atmosphere. Cementation by tungsten is not so effective as cementation by chromium in increasing the resistance of steel surfaces to oxidation.

M. Cook.

Cementation of copper and its alloys by aluminium. L. Guillet (Compt. rend., 1926, **182**, 1447—1449; cf. B., 1926, 588).—Copper, brasses, bronzes, German silver, and copper-nickel alloys were cemented at 700° and 800°, using a powdered aluminium-copper alloy (20% Al) mixed with 5% of ammonium chloride as the cementing material. With copper the penetration is 0·25 mm. after 24 hrs. at 700°, the cemented layer consisting of the α Cu-Al solid solution, but at 800° the penetration is greater and both α and β solid solutions are formed. The surface layer is harder than the copper, and in the specimen cemented at 800° it can be further hardened by quenching, when the martensitic structure is produced. With brasses (60 and 67% Cu) the penetration is greater, and the surface layer containing the α and β brass constituents is harder than the original metal, but little affected by quenching. With a bronze containing 84% Cu, and a German silver (Cu 60%, Ni 20%) the cemented layer is softer than the original metal, but with cupro-nickel (20% Ni) and a bronze containing 90% Cu the cemented layer is the harder. With the bronzes the surface layer consists of the $\alpha+\beta$ or martensitic structure, but special constituents are formed with the nickel alloys. The presence of the ammonium chloride is essential for good penetration.

W. Hume-Rothery.

Determination of small amounts of bismuth in copper. C. O. Jones and E. C. Frost (Ind. Eng. Chem., 1926, **18**, 596).—Good results were obtained by the method in which potassium iodide and sulphurous acid are added to a sulphuric acid solution of bismuth sulphide and the colour compared with that of a standard bismuth solution similarly treated. The bismuth sulphide, previously separated from copper sulphide, is first dissolved in nitric acid, the solution evaporated with sulphuric acid, and any lead sulphate which separates is removed.

B. W. Clarke.

Action of nitric acid on aluminium. S. Uchida and K. Sasaki (J. Soc. Chem. Ind. Japan, 1926, **29**, 93—94).—The action of pure nitric acid on aluminium (99·51%) is of a purely chemical nature, not being influenced by diffusion, especially at low temperature. The solution velocity is doubled for a rise of 10°. The optimum solution velocity is obtained with 5—6 N-acid and at higher concentrations, *e.g.*, above 20N, the velocity is almost zero. Addition of a small amount of carbamide has no influence on the solution velocity, showing that nitric acid acts as an acid

toward aluminium, and that the nitrogen oxides have no effect on the solution velocity. The oxide film formed on aluminium has no acid-proof character. There is a close relation between the hydrogen-ion concentration and the solution velocity. Addition of sulphuric acid, ammonium nitrate, chlorine, etc. has no special effect on the action. K. KASHIMA.

Mechanism of ageing process in aluminium alloys. W. FRAENKEL (Z. Metallk., 1926, 18, 189—192).—To endeavour to elucidate the mechanism of the ageing process in aluminium alloys the change of electrical resistance of quenched alloys with time of ageing has been investigated. In the case of aludur (aluminium with magnesium and only small amounts of iron and silicon) the resistance rises during a short period, then falls much more rapidly over several days; the resistance-time curves for 20° and 167° are practically parallel. Aeron (containing copper and a little silicon but no magnesium) behaves differently in that the resistance falls rapidly at first, then more slowly. The resistance of Al_2Zn_3 (21% Al, 79% Zn) increases by 10—12% in the first few minutes after quenching, then falls rapidly at first, finally more slowly until it is only 66% of that of the quenched alloy; these phenomena are greatly retarded by the presence of even small quantities of magnesium, 0·1% Mg extending the time required for completion of the change from a few days to more than 3 months. In view of these results it is suggested that two changes take place during the ageing of aluminium alloys, one associated with an increase of electrical resistance and the other (retarded by the presence of magnesium) with a decrease in the resistance. In the case of duralumin, which contains magnesium, only the first change takes place at the ordinary temperature within a measurable time, whereas in alloys free from magnesium this change takes place spontaneously on quenching and the second change only after ageing at 100—200°. This theory is in accord with the fact that alloys free from magnesium are stronger immediately after quenching than those containing magnesium, *e.g.*, the tensile strength of quenched duralumin is 16—18 kg. per sq. mm., whilst that of aeron is 28—30 kg. On the other hand the 8% zinc-aluminium alloy without magnesium is no harder after quenching than the same alloy containing magnesium, so that the theory does not appear to explain all the known facts. A. R. POWELL.

Analysis of boron alloys. N. TSCHISCHEWSKI (Ind. Eng. Chem., 1926, 18, 607—608).—The alloy is dissolved in sulphuric acid, oxidised by hydrogen peroxide, and the metals present are precipitated by electrolysis. The boric acid in the resulting solution is determined by titration with caustic soda free from carbon dioxide, using phenolphthalein as indicator, after neutralising the residual sulphuric acid with caustic soda, using methyl-orange as an indicator, which is not affected by boric acid. The results are accurate. B. W. CLARKE.

Contribution to the study of ternary alloys. [Aluminium–magnesium–cadmium and system potassium chloride–barium chloride–magnesium chloride]. J. VALENTIN (Rev. Mét., 1926, 23, 209—218, 295—314; cf. B., 1925, 135).—The ternary eutectic of the aluminium–magnesium–cadmium system, m.p. 395°, contains in atomic per cent., 45 of magnesium, 17·5 of aluminium, and 37·5 of cadmium. In alloys rich in aluminium, the replacement of small percentages of magnesium by equivalent amounts of cadmium does not appreciably affect the mechanical properties, and in magnesium-rich alloys the replacement of small amounts of aluminium by cadmium is also without effect. The constitution of the system, potassium chloride–barium chloride–magnesium chloride, which contains three ternary eutectics, has been studied by thermal analysis and the ternary diagram as well as sectional diagrams are given. M. COOK.

Removal of iron from copper and nickel mattes. B. BOGITCH (Compt. rend., 1926, 182, 1473—1475).—Since the heat of formation of ferrous oxide is greater than that of cuprous oxide, it is usually thought that the loss of copper or nickel from the mattes during removal of iron is due to mechanical entanglement in the slag. To test this, samples of copper and nickel mattes were powdered, mixed with sand and sodium sulphate, and then fused, the sodium sulphate acting as oxidiser. The resulting slag was then analysed for entangled globules of matte, and for copper or nickel present as oxide in the slag. Both copper and nickel are oxidised, and in increasing proportions as the removal of iron becomes more complete. For the greater part of the process the loss of nickel is less than that of copper, in spite of the fact that nickel oxide has a higher heat of formation than copper oxide; this is because of the difference in the heats of decomposition of cuprous and nickel sulphides, as a result of which the molten nickel decomposes sulphur dioxide whilst copper does not. On the other hand, the removal of the last traces of iron is more difficult from nickel than from copper mattes. The more acid (and hence more viscous) the slag, the greater is the loss by mechanical entanglement, but this does not affect the relative amounts lost by oxidation. In practice not more than 4—5% of the copper should be lost in this way. W. HUME-ROTHERY.

Pyrites roasting. HÜTTIG and LÜRMANN.—See VII.

Theory of electric arc furnace. DE LOISY.—See XI.

Corrosion of metals by insulating pastes. REINER.—See XI.

PATENTS.

Refining crude iron. F. BURGERS (U.S.P. 1,578,009, 23.3.26. Appl., 12.3.25).—Crude iron is melted and conveyed to a refining furnace into which it is introduced at a point near the vertical axis and below the upper limit of incandescence.

The fuel supplied to the furnace is preheated by waste gases and is charged through a shoot. M. Cook.

Producing iron coke. H. E. Wetherbee (U.S.P. 1,581,898, 20.4.26. Appl., 13.6.24).—Ferrous or ferric hydroxides, prepared by treating an iron solution with caustic soda, are washed and mixed with a reducing agent, *e.g.*, finely-powdered coal or charcoal, or crude oil having an asphaltic base, and heated gradually to expel moisture. When all the combined moisture has been driven off and the residue consists of iron oxides and the reducing agent, the mass is heated more strongly in a reducing atmosphere, in which it must also be cooled. Reduction starts at 200° and is complete at 550°. E. S. Kreis.

Producing iron directly out of iron ore. H. G. Flodin and E. G. T. Gustafson, Assrs. to H. G. E. Cornelius (U.S.P. 1,585,240, 18.5.26. Appl., 7.4.25).—A charge of oxide ore and carbon in a proportion necessary to effect reduction and carbonisation of the metal is fed into a furnace together with another charge of higher carbon content, the carbon in the resulting metal being controlled by suitably proportioning the two charges. W. Cook.

Manufacture of uranium. Westinghouse Lamp Co., Assees. of J. W. Nearden (E.P. 246,147, 13.1.26. Conv., 13.1.25).—Uranium powder is produced by reducing a uranium salt, *e.g.*, a halide, or potassium uranium fluoride, by means of calcium in the presence of calcium chloride. The powder is then pressed into the desired shape and heated in an inert atmosphere until homogeneous and ductile. C. A. King.

Separating antimony and lead. E. C. R. Marks. From Amer. Smelting and Refining Co. (E.P. 251,736, 17.3.25).—Lead containing antimony in substantial quantity, *e.g.*, above 7%, is heated at 650—930° under oxidising conditions, antimony oxide being recovered as a fume. A layer of lead oxide may be maintained on the surface of the bath during the operation. Any tin present may be removed first as a slag and after volatilisation of antimony, copper separates as a slag on the surface of the cooling lead. C. A. King.

Copper[-magnesium-cadmium] alloys. O. von Rosthorn (E.P. 251,906, 19.1.26).—In the preparation of alloys of copper, magnesium, and cadmium, molten copper is deoxidised by adding a small quantity of an alloy containing Cu 90%, Cd 10%, and a desired amount of a previously prepared auxiliary alloy (Cu 16·5, Mg 3·25, Cd 5 pts.) encased in a copper container is pushed to the bottom of the molten bath and allowed to alloy with the deoxidised copper. The proportion of magnesium may be varied by introducing magnesium before the auxiliary alloy. C. A. King.

Manufacture of alloys containing tungsten carbide. Gen. Electric Co., Assees. of Patent Treuhaud Ges. für Elektrische Glühlampen m.b.h. (E.P. 251,929, 2.9.25. Conv., 7.5.25. Addn. to 213,524; B., 1924, 752).—A sintered hard alloy for tools consisting principally of tungsten monocarbide contains 10—20% of an auxiliary metal of lower melting point, such as iron, cobalt, or nickel. During the preparation of the tungsten-carbide, the temperature is regulated so that the small particles adhere only loosely. C. A. King.

[Alloy for] thermocouple casing. R. P. Brown, Assr. to Brown Instrument Co. (U.S.P. 1,581,380, 20.4.26. Appl., 30.11.25).—A thermocouple casing resistant to the corrosive action of molten lead and zinc is made from an iron-chromium alloy, containing, for example, C 0·10, Si 0·20, Mn 0·30, Cr 13·00, Fe 86·40%. E. S. Kreis.

Manufacture of ferrochromium alloy. R. Wild, Assr. to Rustless Iron Corp. of America (U.S.P. 1,586,590, 1.6.26. Appl., 5.2.23).—See E.P. 197,733; B., 1923, 662 a.

Manufacture of ferro-alloys, particularly ferrochromium alloys. R. Wild, Assr. to Rustless Iron Corp. of America (U.S.P. 1,586,591, 1.6.26. Appl., 5.2.23. Conv., 1.3.22).—See E.P. 198,423; B., 1923, 781 a.

Manufacture of unstainable irons and steels. R. Wild, Assr. to Rustless Iron Corp. of America (U.S.P. 1,586,592, 1.6.26. Appl., 4.5.23).—See E.P. 198,633; B., 1923, 782 a.

Casting metals [nickel-iron alloys]. J. H. White, Assr. to Western Electric Co. (U.S.P. 1,586,871, 1.6.26. Appl., 29.12.24).—See E.P. 241,756; B., 1926, 17.

Steel alloy. B. Strauss, Assr. to F. Krupp A.-G. (U.S.P. 1,587,614, 8.6.26. Appl., 17.6.23. Conv., 2.8.22).—See E.P. 201,915; B., 1924, 60.

Recuperation of metals contained, in the form of silicates, in waste products [slags]. Le Cuivre Natif (E.P. 230,471, 5.3.25. Conv., 7.3.24).—See U.S.P. 1,544,493; B., 1925, 678.

Process of treating metals and composition therefor. E. C. R. Marks. From Chicago Crucible Co. (E.P. 253,402, 14.10.25).—See U.S.P. 1,568,271; B., 1926, 283.

[Incorporating graphite with] bearing material [metal]. A. A. Crimp (E.P. 253,022, 19.1.26).

XI.—ELECTROTECHNICS.

Theory of the electric arc furnace according to recent work. E. de Loisy (Rev. Mét., 1926, 23, 253—268). The subject is treated mathematically and the recent work by P. Bergeon (IVe Congrès Chim. Ind., Bordeaux, Juin, 1924; Soc. Franç. Electricians, Oct., 1925) and E. Riecke (Ber. No. 102, Fachausschüsse Ver. Deuts. Eisenhüttenleute) is reviewed in detail, the latter dealing with furnaces as used for steel melting. M. Cook.

Corrosion of metals by insulating pastes. S. REINER (Z. angew. Chem., 1926, **39**, 588—591).—Insulating oil heated in absence of air is partially dehydrated, with no change in the acid value. The insulating properties are thereby improved. Mineral oils alone have no solvent action on copper, lead, or iron, but the resins with which they are mixed to form insulating pastes, dissolve at 100° up to 0·38% of copper, 1·0% of lead, and 2·0% of iron; in the last case, an increase in the acid value ascribed to polymerisation of the resin is observed. Deterioration of insulating pastes is ascribed to the solvent action of the resinous constituent upon metals, and to water, the presence of which facilitates oxidation of the oils. L. M. CLARKE.

Preparation of luminescent discharge tubes of helium. J. RISLER (Compt. rend., 1926, **182**, 1457—1459).—Particulars are given of the removal of all impurities in the filling of discharge tubes with helium. When the absorbed gases have been removed from the glass and electrodes, impurities may still be introduced with the helium, but this may be prevented by passing the gases over absorptive wood charcoal at —78° (solid carbon dioxide and acetone). An alternative method consists in connecting the discharge tube to a vacuum pump and providing an additional electrode consisting of a small metal crucible containing an alkali or alkaline-earth metal. The tube is evacuated, a discharge passed, and the helium admitted, when all the impurities are absorbed by the metal vapour which is liberated. The following figures are given for the volumes of gases absorbed by 1 g. of wood charcoal at —78°, and by 1 g. of sodium and of calcium under the above conditions. Hydrogen 55, 125, and 130 c.c.; nitrogen 95, 135, and 140 c.c.; carbon dioxide 145, 175, and 165 c.c.; helium, 5, 4, and 4 c.c.; neon, 6, 6, and 5 c.c. respectively. Charcoal absorbs 130 c.c. of oxygen at —78°.
W. HUME-ROTHERY.

PATENTS.

High-frequency electric induction furnace. C. R. BURCH, N. R. DAVIS, and METROPOLITAN-VICKERS ELECTRICAL CO. (E.P. 251,758, 18.4.25).—In a high-frequency electric induction furnace in which the inductor consists of a tube and has a cooling fluid circulated through it, the inlet and outlet for the cooling liquid are arranged at equipotential points. This may be done by winding a doubled tube to form a spiral. Electrical connexions are made to the two ends of the spiral and fluid connexions to the tube ends. Other methods consist in winding a number of turns on a tube and then winding in the reverse direction, or by providing a high-frequency choke coil made of tubing and connected electrically in parallel with the inductor tube. Arrangements for automatically regulating the current energy may be incorporated such as the insertion of a Venturi tube and mercury gauge which makes or breaks contact with a controlling battery, or by means of a thermostat in the cooling fluid. C. A. KING.

Method of growing crystals [with piezo-electric properties]. A. M. NICOLSON, Assr. to WESTERN ELECTRIC CO. (U.S.P. 1,578,677, 30.3.26. Appl., 30.9.24).—The patent relates in general to the production of crystals possessing piezo-electric properties and an "hour-glass" structure, and, in particular, to the formation of such crystals of Rochelle salt. Seed crystals are used having three axes "*a*," "*b*," and "*c*" (the principal axis) mutually perpendicular. An "hour-glass" structure is produced when crystals are grown from such seed crystals placed with the axes "*c*" and "*b*" in a horizontal plane; growth proceeds laterally but is arrested upwards along the axis "*a*" The thickness of a seed crystal along its "*a*" axis should be 4/10 of that along its "*b*" axis. The crystals are grown as follows: Powdered Rochelle salt is placed in contact with water at a temperature not exceeding 40° (38—40°) until a constant density of d^{38-50} 1·39 is obtained. Water at the same temperature is stirred into the saturated mother liquor until the density is d^{38-50} 1·37—1·38, after which the solution is filtered into glass crystallising dishes maintained at 38—40°. Seed crystals are added as described above to the dishes which are then kept at 20° for 10—12 hrs. R. B. CLARKE.

Alumina or aluminium sulphate. (Addn. to F.P. 586,684).—See VII.

Mixtures of hydrogen and nitrogen (G.P. 427,542).—See VII.

Electrodeposition of organic materials (U.S.P. 1,580,795).—See XIV.

XII.—FATS; OILS; WAXES.

Marine animal oils. M. TSUJIMOTO (J. Soc. Chem. Ind. Japan, 1926, **29**, 71—75).—The author has examined the following oils: three samples of lamprey oil, sturgeon (*Acipenser mikadoi*) liver oil, "tora-fugu" ("tiger globe fish," *Spheroides rubripes*) liver oil, dolphin (*Delphinus longirostris*) liver oil, and "hotaru-ika" ("luminous cuttle-fish," *Watasenia scintillaus*) oil. The densities, acid, saponification, and iodine values, index of refraction, amount of unsaponifiable matter and of ether-insoluble bromides of mixed fatty acids, and colour reactions of unsaponifiable matter with sulphuric acid in carbon disulphide and acid earth in benzol are given in a table. The "tora-fugu" liver oil deposited a considerable quantity of solid fat. The unsaponifiable matter of lamprey and dolphin oils contains a large amount of a resinous substance, which gives the colour reactions very distinctly. The unsaponifiable matter of "hotaru-ika" oil consists chiefly of cholesterol. K. KASHIMA.

Constitution of the unsaturated acid $C_{14}H_{26}O_2$ from sperm oil. M. TSUJIMOTO (J. Soc. Chem. Ind. Japan, 1926, **29**, 102—104).—The author has examined the oxidation products of the tetradecenoic acid, $C_{14}H_{26}O_2$, from sperm oil (B., 1923, 276 A), by the method of Armstrong and Hilditch (J.S.C.I.,

1925, 45 T and 108 T). Nonoic acid and an acid, probably glutaric acid (though the m.p. is somewhat low) were isolated; valeric and azelaic acids were not detected. The constitution of the decenoic acid, $CH_3\cdot[CH_2]_7\cdot CH{:}CH\cdot[CH_2]_3\cdot CO_2H$, determined by the ozone method (B., 1925, 856), is thus confirmed.

K. Kashima.

Lower acids of the oleic series in "tsuzu" and "kuromoji" seed oils. M. Tsujimoto (J. Soc. Chem. Ind. Japan, 1926, **29**, 105—108).—Oils from seeds of "tsuzu" (*Tetradenia glauca*) and "kuromoji" (*Lindera hypglauca*) had the following characteristics: orange-yellow, dark greenish-yellow, d_4^{15} 0·9452, 0·9394; acid value 13·4, 38·8; saponif. value 238·3, 222·5; iodine value 79·4, 69·1; n^{20} 1·4670, 1·4727; Reichert-Meissl value 1·30, 1·49; and unsaponifiable matter 2·45%, 4·12%. A dodecenoic acid (probably identical with Iwamoto's linderic acid, B., 1921, 856 A) is present in large amount in the oils, and a tetradecenoic acid is present in small amount—in very small amount in the "kuromoji" seed oil. The tetradecenoic acid isolated from tsuzu oil is different from the acid obtained from sperm, dolphin, and tôhaku oils (cf. preceding abstract), being distinguished by its higher m.p. (about 20°). It is a crystalline solid at the ordinary temperature.

K. Kashima.

Formation of unsaturated solid *iso*-acids during the hydrogenation of fatty oils. II. Presence of *iso*-acids in hardened chrysalis oil. S. Ueno and N. Kuze (J. Soc. Chem. Ind. Japan, 1926, **29**, 75—77).—Chrysalis oil (d_4^{100} 0·8683, n^{50} 1·4649, acid value 6·3, saponif. value 191·2, iodine value 128·5) was hardened at 140°. 160°, and 180° respectively, using a nickel catalyst. The fatty acids of the hardened oils were separated into solid and liquid acids by the lead salt–ether method. Chrysalis oil appears to contain originally a small amount of unsaturated *iso*-acids. Solid unsaturated *iso*-acids are formed during the hydrogenation, the amount increasing with rise of hydrogenating temperature.

K. Kashima.

Influence of saturated fatty acids on the value of the Boemer number for lard. F. J. F. Muschter and R. Smit (Chem. Weekblad, 1926, **23**, 284—285).—The case is quoted of a crude lard found to have a Boemer value (cf. B., 1922, 431 A) greater than 71, but which yielded by refining a neutral lard having a value below 71. Addition of free fatty acids to the neutral lard gave products having values up to 72·2.

S. I. Levy.

Polymerised linseed oil. K. H. Bauer (Farben-Ztg., 1926, **31**, 2130—2131).—A sample of polymerised linseed oil was heated until it attained the consistency of rubber, when it gave the following figures: saponif. value 206, acetone-soluble matter 37·7%, chloroform-soluble matter 44·7%. Fatty acids obtained by saponification had acid value 200, iodine value 98·7, and hexabromide value 0. The mol. wt. of the fatty acids was determined cryoscopically in benzene and by Rast's camphor method. Mol. wt. in benzene 685·0, in camphor 321·0. Fatty acids obtained from the acetone-soluble portion had acid value 197, iodine value 100·6, mol. wt. in benzene 453·0, and in camphor 244·0. Fatty acids obtained from the chloroform-soluble portion had acid value 193·7, iodine value 93·3, mol. wt. in benzene 447·0, and in camphor 241·0. The values in camphor are mono- and those in benzene bi-molecular, a fact which throws light on discrepancies among investigators dealing with polymerisation of oils.

S. S. Woolf.

Polymerisation of tung oil. H. Wolff (Z. angew. Chem., 1926, **39**, 767—770; Farben-Ztg., 1926, **31**, 2235, 2292—2294; cf. B., 1925, 556).—Further evidence is adduced from the author's own researches and from those of other investigators to show that polymerisation is not the sole cause of the thickening of tung oil by heating. Thus, agitation of the oil with a very small proportion of hydrochloric acid (*d* 1·125) at 30—40° induces thickening at a rate comparable with that caused by heating at 200°, whilst no appreciable change takes place in the iodine value. This appears to indicate that certain colloidal constituents of the oil are polymerised by this treatment and that the thickening caused by heat is due to two independent phenomena, namely, coagulation of colloids and chemical change comprising polymerisation, decomposition of some of the glycerides, and changes in the configuration of the elæostearin glycerides.

A. R. Powell.

Properties of sodium naphthenate used in soap industry. Y. Kawakami (J. Soc. Chem. Ind. Japan, 1926, **29**, 64—67).—The naphthenic acid used had b.p. 195—200° /10 mm., n_D^{22} 1·4812, d_{15}^{22} 0·9605, neutralisation value 228·0, mean mol. wt. 246·5. The detergent action and lathering power of the sodium salt of the acid were slightly inferior to those of sodium palmitate and mixed toilet soap. The gelation capacity of sodium naphthenate was extraordinarily large. The sodium salt of a lower naphthenic acid required more salt than coconut oil soap in the salting-out process, but 100 g. of a 1% solution of a sodium salt of a higher naphthenic acid salted out with 4 g. of salt at 100°, whereas in the same conditions, coconut oil soap required 11 g. of salt and sodium palmitate 6 g. Sodium naphthenate is hygroscopic in moist air.

K. Kashima.

Patent.

Recovery of oils and fats from finely divided bleaching materials or the like. Harburger Eisen- u. Bronzewerke A.-G. (G.P. 426,712, 7.7.21).—The oil or fat contained in used decolorising carbon or fuller's earth may be recovered by heating the mass with a dilute solution of alkali hydroxide at 100° in an autoclave fitted with a stirring device, then raising the temperature and pressure after addition of a dilute salt solution, and finally allowing the mixture to cool without agitation. The alkali and salt treatments cause the surface tension between the solid particles and the oil or fat to be so far reduced that the two separate, the carbon or fuller's earth settling to the bottom of the vessel

and the oil or fat forming a layer on the surface of the solution. A. R. POWELL.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

So-called black and white content of pigments in the Ostwald colour-system. C. SCHAEFER (Physikal. Z., 1926, **27**, 347—353).—Ostwald's absolute system of colours (A., 1916, ii, 205 ; 1917, ii, 281) is criticised (see also Kohlrausch, Physikal, Z., 1920, **21**, 423, 473). It is shown experimentally that insertion of dark sectors in a colour-circle merely reduces brightness in a manner which is exactly counterbalanced by increased intensity of illumination. This result is in opposition to Ostwald's theory. Further theoretical and experimental arguments are adduced against the theory that the three terms pure colour, white, and black can express colour quantitatively. R. A. MORTON.

Relation between the number and size of the particles and the light absorption of graphite suspensions. F. HEBLER (Z. angew. Chem., 1926, **39**, 741—744).—The light absorption of colloidal suspensions of graphite in water or water-glycerin mixtures increases at first with the degree of dispersion in a ratio approximately proportional to the increase in the number of particles corresponding with the greater dispersion. The absorption by colloidal suspensions containing particles of an average size of 2·5 μ is, however, about 50% less than the theoretical calculated from the linear law, but the maximum absorption of light is not reached at this grain size. A. R. POWELL.

Evaluation of [water-]paint adhesives. H. WAGNER (Farben-Ztg., 1926, 31, 2132).—Two types of test are indicated. The first, mainly economic, deals with adhesive strength and effective cost. Trial mixtures are made up with calcium carbonate as pigment, and the quantities used for satisfactory binding are substituted in empirical formulæ giving comparison figures. Mixtures are then made up using pigments of acid properties and their behaviour on painting gives information as to adsorption, peptisation, reversibility, alkalinity, refractive power, etc., properties which bear on applicability, adhesion, permanence, water-resistance, opacity, etc. Glass plates coated for this second test may be used as negatives to obtain permanent records for comparison of various tested products. S. S. WOOLF.

Micrography of oil and varnish films. E. STERN (Farben-Ztg., 1926, 31, 2129—2130).—The examination of the microstructure of films of varnish and paint-media is recommended as a simple form of accelerated testing, information as to quality and durability being afforded by the alteration of the original microstructure during the drying and hardening period, and the ageing and deterioration of the dried film. The development of suitable etching technique should reveal characteristic behaviour in films containing reversible and irreversible emulsions, linseed and tung oils, cellulose esters, etc. S. S. WOOLF.

American colophony. P. LEVY and H. RAALF (Ber., 1926, **59**, 1302—1310).—Oxidation of crude American colophony in cold, aqueous alkaline solution by potassium permanganate gives formic, acetic, propionic, and *iso*butyric acids, together with r-*tetrahydroxyabietic acid*, m.p. 207·5—209°, and *l*-tetrahydroxyabietic acid, m.p. 251—252°, $[\alpha]_D^{15}$ —41·64° in alcohol. The presence of two ethylenic linkings in abietic acid is therefore established. H. WREN.

Polymerised linseed oil. BAUER.—See XII.

Polymerisation of tung oil. WOLFF.—See XII.

PATENTS.

Colours capable of being fixed by heat. O. VAN CUYCK (E.P. 251,866, 29.10.25. Conv., 8.5.25).—Pigments are incorporated with nitre and gum-resin, and the ground products are used in the form of pastels or as liquid fresco-paints. The colours are so easily fusible that they can be glazed or vitrified by the application of a temperature insufficient to cause deterioration of the paper, textile, etc., on which they are applied. For example, Venetian red is obtained by drying a watery paste of 10 pts. of iron oxide, 2 pts. of nitre, 1 pt. of glycerol, and 4 pts. of flowers of sulphur. The ground residue is fused with 10 pts. of kauri gum and 2 pts. of nitre, and the cooled mass ground and pressed into crayons or tablets. Other resins such as white mastic gum may replace the kauri gum. S. S. WOOLF.

Resinous compositions. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of L. V. ADAMS (E.P. 235,595, 13.6.25. Conv., 13.6.24).—A condensation product of glycerol and phthalic anhydride (referred to as a "glyptal") is incorporated with a drying oil in any proportions by heating to about 200° in contact with a solvent of high b.p., *e.g.*, benzyl benzoate (b.p. 323—325°). The glyptal is used in its initial soluble form and during the heating process it becomes converted into the intermediate less fusible and less soluble form. The high-boiling solvent may be removed from the product by distillation, leaving a viscous mass which dissolves in solvent naphtha etc. to produce a varnish. The glyptal in the product is readily converted into the final insoluble and infusible form, and a tough, flexible, infusible film, of good adhesive properties and resistance to heat is obtained. Semi- and non-drying oils may be substituted for the drying oil, while other solvents of only moderately high b.p. may be used as dispersing agents, reflux condensation being adopted in such cases to avoid excessive loss. S. S. WOOLF.

Process of manufacturing acid-proof and water-proof black ink. J. INOUYE (E.P. 253,368, 30.6.25).—See F.P. 600,390 ; B., 1926, 596.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Origin of the [X-ray] interferences in the stretching of rubber. E. A. HAUSER (with H. MARK) (Gummi-Ztg., 1926, **40**, 2090—2092).—

Further investigation of the X-ray interferences exhibited by stretched rubber indicates that the phenomena at high extension are due in part to nuclei of the magnitude $(C_5H_8)_8$. At lower extensions the phenomena are produced by clusters containing approximately 2000 (C_5H_8) groups; whether these are to be regarded as crystals or as aggregates will be dependent on the amplitude of vibrations of the particles. These aggregates are already existent in a swollen condition in the unstretched rubber, but give no definite interference phenomena because they have a vibration amplitude too large to give rise to interference phenomena, the state of their " crystal lattice " being comparable with that of strongly heated salt. Under stress, however, part of the distending medium is reversibly squeezed from the aggregates so that these then exhibit distinct interference behaviour. This explanation accords with the observation that the position of the interferences is independent of the degree of extension while their intensity increases almost droportionately with this factor. D. F. Twiss.

Determination of sulphur in vulcanised rubber. E. Kahane (Caoutchouc et Gutta-percha, 1926, **23**, 13,154—13,155).—For determination of its total sulphur content, vulcanised rubber is commonly oxidised with brominated nitric acid followed by fusion (cf. Tuttle and Isaacs, B., 1915, 436). In the absence of such mineral ingredients as barium carbonate, the subsequent fusion may conveniently be omitted. By adopting similar procedure for the determination of the total sulphur and mineral sulphur, the difference between the two results (necessary for calculation of the degree of vulcanisation) is the same as would be obtained by the longer method. It may be necessary to wash the barium sulphate precipitate with aqueous ammonia to remove oxidation products of the rubber. D. F. Twiss.

Patent.

Electrodeposition of organic materials [rubber]. S. E. Sheppard and L. W. Eberlin, Assrs. to Eastman Kodak Co. (U.S.P. 1,580,795, 13.4.26. Appl., 8.6.25).—When organic materials are electrolytically deposited from emulsions or suspensions, the deposition may be hastened by the use of a high current density, but this causes the evolution at the anode, of gases, particularly oxygen, which interfere with the formation of an even deposit and cause pits and weak spots. This can be avoided by having in solution a reducing agent which is indifferent to the material deposited, for instance, alkali sulphite, hyposulphite, or thiosulphate with rubber emulsions. Organic reducing agents may be used; if a substance which will oxidise to a coloured substance is used the deposit will be coloured.
E. S. Kreis.

XV.—LEATHER; GLUE.

Nature of vegetable tannage: Tanning with mixtures of gallotannin and quinone. A. W. Thomas and M. W. Kelly (Ind. Eng. Chem., 1926, **18**, 625—626).—The fixation of tannin by hide powder from solutions of gallotannic acid and benzoquinone respectively was determined in solutions of varying p_H value. The quinone curve showed a maximum at p_H 10·5. The curves for mixtures of gallotannic acid and benzoquinone in different proportions were also determined and show increasing amounts of fixation in the alkaline range of p_H 8 to 12, similar to the typical curve for vegetable tanning (cf. B., 1923, 1235 A). In the region of p_H 8 to 5 the quinone retards the tanning, thus acting like a non-tan. The results support the view that the tanning action of vegetable tanning extracts in the range p_H 8 to 12 is due to substances of the nature of quinones. D. Woodroffe.

Processes in chrome tanning. S. Hilpert and E. Schlumberger (Z. angew. Chem., 1926, **39**, 637—640).—Samples of gelatin were shaken for 3 hrs. with a large excess of solutions of basic chrome tanning liquors of different concentrations. The product was analysed in each case for unchanged gelatin. When this was nil the tannage was counted as 100%. The p_H value and concentration of those liquors which gave 100% tannage were determined and the results plotted. The logarithm of the chromium concentration was inversely proportional to the p_H value whether the liquors were clear or turbid or whether green or violet. A mixture of a molten 16% gelatin jelly and a 20% solution of chromium sulphate, which was almost at the precipitation point, set to a mass from which a large amount of water could be decanted. Chrome tanning is influenced more by the condition of the pelt than by the chrome liquor. Swelling is due to the hydration of the primary amino-groups. The lower the p_H value, the greater the amount of hydration and consequently the less possibility for the tannin molecules to link up with the amino-groups. The optimum speed of tanning is therefore at the isoelectric point. It is impossible to produce normal chrome liquors with a p_H equal to that of the isoelectric point since it is on the wrong side of the precipitation point. The isoelectric point of proteins can be changed by additions of acid and by neutral salts. The tanning intensity does not depend on the basicity of the chrome tanning solution but on its p_H value and is at a maximum when the greatest possible degree of dehydration of the hide protein is attained. D. Woodroffe.

Properties of various glues. T. Araki (J. Soc. Chem. Ind. Japan, 1926, **29**, 84—92).—The author has compared the properties of various samples of Japanese and foreign glues, and studied the relations between various properties and the quality of the glue. The adhesive strength was found to be generally proportional to the jelly strength except that a few glue solutions of high concentrations gave high values. Solutions of 1 pt. of glue in 2 pts. of water such as are generally used for strong joints showed very high adhesive power, and in the strength test the walnut test-pieces were stripped off whilst the glue surfaces remained un-

affected. The adhesive powers are greater with straight-grained wood than with cross-grained wood.
K. Kashima.

Unhairing process. Alteration of keratin by alkalis. II. M. Bergmann and F. Stather (Collegium, 1926, 249—256).—See A., 1926, 631.

Patents.

Lime treatment of skins. M. Bourguignon, Assr. to Soc. du Feutre (U.S.P. 1,586,062, 25.5.26. Appl., 7.2.22).—See E.P. 175,620; B., 1923, 367 a.

Quick tannage by means of the one-bath method. Chem. Fabr. Griesheim-Elektron, Assees. of F. Merkel (U.S.P. 1,587,019, 1.6.26. Appl., 18.7.22. Conv., 1.8.21).—See G.P. 414,867; B., 1925, 858.

XVI.—AGRICULTURE.

Properties of humid-tropical and humid-temperate American soils with reference to relations between chemical composition and physical properties. H. H. Bennett (Soil Sci., 1926, **21**, 349—376).—In a study of clay soils occurring in tropical America, two types are distinguished, namely, a friable and a plastic type. The former type is distinguished by a relatively low silica and high sesquioxide content, the molecular ratio $SiO_2 : Al_2O_3+Fe_2O_3$ being generally less than 2. In the plastic type this ratio is greater than 2. Similar conclusions are reached from a consideration of certain clays of the South-Eastern United States.
G. W. Robinson.

Loss of soluble salts in "run-off" water [from soils]. F. L. Duley (Soil Sci., 1926, **21**, 401—409).—Analyses are given of the water running off the surface of different plots in a series of soil erosion experiments in Missouri. The total annual loss of soluble matter varied from 167 to 380 lb. per acre, and fell chiefly on the calcium and sulphur of the soil. Whilst the losses of potassium were relatively small, in some cases they were equivalent to the amount ordinarily applied in potash fertilisers. The losses of nitrogen and phosphorus were comparatively small.
G. W. Robinson.

Determination of incrusted cellulose in soil. N. Bengtsson (Mitt. 279 der landw. Zentralversuchsanst. [Sweden], 1926, Bakt. Abhandl., 37, 1—13; Chem. Zentr., 1926, I., 3355).—For the determination of the cellulose of straw in soil, 20 g. of soil are digested at 100° for 72 hrs. with a solution of 80 g. of sodium hydrogen sulphite in 100 c.c. of water and 200 c.c. of *N*-hydrochloric acid. With sawdust and moss, longer heating is required. The residue is washed and dried at 50°, shaken for 1—2 hrs. with 100 c.c. of Schweitzer's reagent, and the liquid filtered after keeping overnight. The cellulose in 50 c.c. of the filtrate is then precipitated with 80% alcohol, washed successively with hydrochloric acid, 5% ammonia, 2% hydrochloric acid, water, alcohol, and ether, and dried for ½ hr. at 50° and for 1 hr. at 100°. It is then transferred to a platinum crucible, weighed, ignited, and re-weighed. The difference between the two weighings gives the cellulose in 10 g. of soil. A correction must be made for the water in the precipitate before treatment with Schweitzer's reagent, if very accurate results are required. The procedure is slightly modified for the determination of cellulose in lignite.
C. T. Gimingham.

Decomposition of incrusted cellulose in soil I. Straw and sawdust in loamy and sandy soils C. Barthel and N. Bengtsson (Mitt. 300 der landw. Zentralversuchsanst. [Sweden], 1926, Bakt Abhandl., 40, 1—19; Chem. Zentr., 1926, I., 3354).—Experiments with oat straw and sawdust show that the reaction of the soil has even less influence on the decomposition of incrusted cellulose than it has on that of pure cellulose. Oat straw contains a sufficient amount of suitable nitrogen compounds for the cellulose-decomposing bacteria and, consequently, decomposes more rapidly than pure cellulose in sandy soils poor in nitrogen. This also accounts for the rapid breaking down of roots and stubble in the soil.
C. T. Gimingham.

Use of oat straw in a system of soil fertility. R. P. Thomas and H. J. Harper (Soil Sci., 1926, **21**, 393—400).—Addition of oat straw to two types of soil, alone and in combination with red clover, sodium nitrate, and ammonium sulphate, respectively, did not affect the accumulation of nitrates. Straw alone depressed the yield of wheat in pot experiments with one type of soil. No depression occurred with maize on another soil type. It is concluded that large quantities of straw can be returned to soils, if spread on the second growth of clover, without reducing the nitrate content except in the soil which is in immediate contact with the straw.
G. W. Robinson.

Influence of form, soil-zone, and fineness of lime and magnesia incorporations upon outgo of calcium and magnesium. W. H. MacIntire (Soil Sci., 1926, **21**, 377—392).—The author has studied by lysimetric observations the effect of different dressings supplying lime and magnesia and of different modes of application on the loss of these constituents by drainage. The rate of loss of calcium during four years after incorporation of the dressing with the surface zone was not increased by more than 6·8% of the added calcium. Greater losses were observed when the dressings were incorporated with lower zones. Losses of magnesium were only slightly increased by surface incorporation of limestone or dolomite, but incorporation of the latter material with the subsoil resulted in considerably increased losses. Total calcium-magnesium leachings were uniformly greater when the added dressings were incorporated with the subsoil and were approximately the same for additions of calcium hydroxide, 80- to 200-mesh limestone, and 80- to 200-mesh dolomite respectively.
G. W. Robinson.

Decomposition of farmyard manure in soil and its utilisation by plants. M. Bach (Landw.

Vers.-Stat., 1926, **104**, 245—284).—Samples were taken on cropped and fallowed plots on a variety of soils at intervals after manuring with dung. About 75% of the added carbon is oxidised by the end of a year. The remainder oxidises only slowly. Decomposition is notably slower in chalk soils than in sand, clay, and slatey soils. The whole of the pentosans added with the manure disappear within a year, whereas decomposition of the lignin proceeds much more slowly. The amounts of both total and easily soluble nitrogen show a considerable decrease soon after manuring, due to denitrification and loss by drainage. The percentage of available phosphoric acid remains approximately constant; available potassium shows a gradual decrease. On plots cropped with sugar beet and rye, about 20% of the added nitrogen, about 20% of the phosphoric acid, and about 50% of the potassium were utilised by the plants in the two years following manuring.

C. T. GIMINGHAM.

Solubility of soil potassium and response to potassium fertilisers. O. ENGELS (Ernährung d. Pflanze, 1925, **21**, 172—178; Chem. Zentr., 1926, I, 213).—The solubility of the potassium compounds of the soil in 10% hydrochloric acid or in 1% citric acid is no trustworthy indication of the need or otherwise for potassium fertilisers. G. W. ROBINSON.

Vegetation experiments with sericite as a source of potassium. E. BLANCK and F. ALTEN (Landw. Vers.-Stat., 1926, **104**, 237—243).—Vegetation experiments with mustard grown in sand give no support to the view that the potassium in sericite, a potash-mica, can be utilised by plants. No significant difference in yield was observed between pots receiving no potassium and those receiving sericite.

C. T. GIMINGHAM.

Promoloid asahi [colloidal magnesium silicate. E. BOTTINI (Annali Chim. Appl., 1926, **16**, 29—39).—The "promoloid asahi" used was a milky, alkaline liquid containing 88·82% of water (determined at 100°), 2·09% of water of crystallisation and combination (at red heat), 2·03% of magnesia, 6·94% of silica, and 0·12% of ferric oxide; 1000 c.c. required 2·08 g. of sulphuric acid for neutralisation. Experiments were made with artificial soils of different types. Addition of "promoloid" diminished the permeability, with all kinds of soil. It had little effect on the capillarity of sandy and calcareous soils, but with clay and humic soils it increased the capillarity considerably, favouring the evaporation of water. "Promoloid" had no appreciable effect on the drying of the wet soils except in the case of humic soils where it accelerated evaporation. The absorptive power of the soils for ammonium chloride was increased and for sodium nitrate diminished by treatment with "promoloid." R. SANSONE.

Denitrification in oxidising media. E. PARISI (Annali Chim. Appl., 1926, **16**, 40—45).—The losses of nitrogen taking place in the biological purification of sewage and maturation of manure during aerobic fermentation, are attributed to the interaction of nitrous acid, formed by oxidation of ammonia, with amino-acids produced by degradation of proteins:

$$R\cdot CH(NH_2)\cdot CO_2H+HNO_2=R\cdot CH(OH)\cdot CO_2H+H_2O+N_2.$$

Ammonium sulphate added to soil submerged in water in a flask in which an atmosphere of oxygen was maintained was decomposed with formation of a considerable quantity of nitrogen. When sufficient sucrose was added to the submerged aerated soil to prevent nitrification the ammoniacal nitrogen remained unchanged. When the quantity of amino-acid present in the soil was not sufficient to destroy the nitrous acid formed nitric acid was produced. By adding sufficient asparagine the whole of the nitrous acid, however, disappeared and the liquid remained free from nitrate. R. SANSONE.

Problem of a proper nutrient medium. D. N. PRIANISHNIKOV and M. K. DOMONTOVITCH (Soil Sci., 1926, **21**, 327—348).—A historical discussion is given of the problem of nutrient solutions for use with sand cultures. In the experimental portion, the authors study the effect of the different ingredients of the Prianishnikov medium in which nitrogen is supplied as ammonium nitrate, in combination with calcium monohydrogen phosphate. In this medium and also in the Hellriegel medium, containing calcium nitrate and potassium dihydrogen phosphate, the greatest buffering effect is at a p_H near the neutral point. Ammonium nitrate is physiologically acid. The effects of different plants on the reaction of complete media containing sodium nitrate, ammonium nitrate, or ammonium chloride with a small amount of potassium dihydrogen phosphate are qualitatively similar. Buckwheat shows the greatest tolerance to the physiological acidity of ammonium chloride.

G. W. ROBINSON.

PATENT.

Artificial drying of crops (E.P. 251,577).—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Chemistry of [sugar] refining by "Norit." P. HONIG (Int. Sugar J., 1926, **28**, 302—305).—Examination of washed Cuban sugar melts after treatment with about 1½% (on the weight of sugar) of "Norit" carbon showed the purity to remain practically unaltered, the colour and the surface tension being principally affected, while ash was adsorbed to the extent of 20—25% of that originally present. After a single application of "Norit," the ash adsorption was about 1·8% of the carbon, calcium, magnesium, and phosphoric acid being principally removed. Organic matters (pectins and pentosans, galactans, wax, and nitrogenous substances) are taken up to the extent of 2·2% of the carbon. Surface tension determinations and laboratory experiments show carbon to be superior to kieselguhr as a filtering aid. J. P. OGILVIE.

Determination of the effect of decolorising carbons on sugar juices by measurement of the surface tension. F. TÖDT (Z. Ver. deuts. Zucker-Ind., 1926, 253—272).—Traube's "stalagmo-

meter" (cf. B., 1888, 42; 1911, 448) is recommended in preference to Du Noüy's apparatus for the determination of the surface tension, especially for practical work in sugar factory control.
J. P. OGILVIE.

Determination of reducing sugars by the picric acid method. F. HERZFELD (Z. Ver. deuts. Zucker-Ind., 1926, 273—292).—In the picric acid method, instead of determining the picramic acid colorimetrically, the extent of the change of colour is estimated by means of a polarisation-photometer, tables giving the extinction coefficients for various sugars having been compiled. In the case of impure factory products, the method is less trustworthy than with fairly pure solutions. J. P. OGILVIE.

Determination of starch by calcium chloride. G. CHABOT (Bull. Soc. chim. Belg., 1926, **35**, 130—131).—The author has examined the method of Mannich and Lenz (Z. Unters. Nahr. Genussm., 1920, **40**, 1) which is based on the fact that starch dissolves when boiled with concentrated calcium chloride solution, yielding a clear solution which can be polarised. He finds that if the calcium chloride used is alkaline, gelatinisation occurs. Clearer solutions are obtained in porcelain than in glass vessels. E. W. WIGNALL.

PATENTS.

Filtration or decolorisation of sugar and other liquors. M. RUSSELL (E.P. 251,749, 4.4.25).—In revivification of animal charcoal and like filtering media by steam as described in E.P. 210,232 (B., 1924, 271) when the filter and filtering medium have reached the temperature of 100° steam may be drawn from the exit end of the filter, compressed to any desired extent in a compressor, and returned through the filter in a closed circuit, steam from the boilers being required only to make good any leakages in the circuit. Charcoal and dust may be separated from the spent steam by passing it through a 160-mesh monel-metal screen or a layer of sand and gravel. G. T. PEARD.

Extraction of juices by diffusion. A SOKOLOV (F.P. 597,408, 29.4.25).—The substances from which the juice is to be extracted, *e.g.*, sugar-beet slices, are first treated in a current of the extracting liquid and then exposed between sieves to the action of a current of hot juice, the direction of which can be periodically altered. C. RANKEN.

Manufacture of starch. J. M. WIDMER, Assr. to PENICK & FORD, LTD. (U.S.P. 1,585,452, 18.5.26. Appl., 26.12.24).—Corn (maize) is steeped, and the grain in disintegrated condition and in presence of water is subjected to separating operations for the removal of the starch. Water used in these operations is sterilised by heat derived from the evaporation of the steep water. Wash water from the starch washing operation is acidified and used together with the sterilised water for the separating operations on material treated subsequently. J. P. OGILVIE.

Preparation of starch solutions not precipitated by basic salts. J. PERL & Co. (Swiss P. 113,725, 19.11.24).—An aqueous starch suspension after warming with very small quantities of acid substances, is heated with caustic alkali in excess until the starch is dissolved, and, finally, the alkali is neutralised by acid.
C. RANKEN.

Treatment of seed of the carob-tree or other plant to extract the gum. SOC. ANON. ETABL. J. F. AUDIBERT (E.P. 241,186, 1.10.25. Conv., 10.10.24).—Carob seeds decorticated by rolling and crushing are heated until of a golden brown colour, and then macerated in 20 times their weight of boiling water, the viscous product obtained being pumped through sieves and cloth filters, and the clear gum dried and pulverised. J. P. OGILVIE.

XVIII.—FERMENTATION INDUSTRIES.

Agave pulp as a source of industrial alcohol. P. BAUD (Compt. rend., 1926, **182**, 1631—1634).—Leaves of *Agave rigida*, var. *Sisalana*, two months after cutting, contain up to 6% of fermentable sugars and 3·5% of non-reducing sugars and gums. Juice expressed from agave pulp after removal of the fibre, contains 5·4% of fermentable sugar and 6·6% of non-reducing sugar including pentosans. The dried pulp contains up to 15% of fermentable sugar, as against the expected value of 40—45%. This variation is ascribed to changes in the composition of leaves at different times of the year. Analyses for cellulose and inorganic salts are also given. 22·4 litres of 92% alcohol were produced from 280 kg. of agave stems. It is considered that the production of alcohol is an economic possibility from five-year-old plantations, not less than 400 hectares in area. M. CLARK.

Toxicity, detection, and determination of methyl alcohol. G. REIF (Z. Unters. Lebensm., 1926, **51**, 262—267).—A case of poisoning by methyl alcohol in spirits is discussed. The toxicity is ascribed to methyl alcohol itself and not to contained impurities. The method of Pfyl, Reif, and Hanner (B., 1922, 78 A) for detecting methyl alcohol by means of guaiacol is adopted for the detection and determination of small quantities of methyl alcohol in spirits. A. G. POLLARD.

PATENTS.

Preparation of citric acid by fermentation. J. Szücs (Austr. P. 101,009, 19.12.23; F.P. 589,936, 3.12.24).—Species of *Citromyces*, *Mucor*, *Aspergillus*, and *Penicillium* chosen after experimental comparison of their power of forming citric acid at relatively low temperatures, are used for the preparation of citric acid by fermentation of molasses. The optimum temperature for *Aspergillus* is 20°, whereby the oxidation of citric acid to oxalic acid is hindered. The molasses may be mixed with phosphates or freed from potassium salts by purification. The citric acid is neutralised by addition of oxide, hydroxide, or carbonate of barium, and the resulting barium citrate decomposed by sulphuric acid.
C. RANKEN.

Manufacture of yeast, especially by the aeration process. VER. MAUTNER'SCHE PRESSHEFEFABR. G.M.B.H., and E. FOULD-SPRINGER (Austr. P. 102,274, 20.9.19).—Yeast and alcohol are prepared from mashes or worts by leading continuously or periodically during fermentation a concentrated solution into the extremely dilute fermenting liquid. The concentration and velocity of the inflowing liquid are such that the consumption of nutriment is not only equalled but exceeded. The fermentation is also carried out so that, in addition to the yeast reproduction, an increasing quantity of alcohol is formed in the fermenting solution.

C. RANKEN.

Obtaining absolute alcohol. J. A. STEFFENS, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,586,717–8, 1.6.26. Appl., 5.4.21).—See E.P. 206,747 and 213,984; B., 1924, 193, 569.

Purification of effluents from breweries, distilleries, etc. (G.P. 426,765).—See XXIII.

XIX.—FOODS.

Relation between the lactic acid content of milk and its loss in dry extract. M. FOUASSIER and G. MAURICE (Bull. Soc. Chim. biol., 1926, **8**, 175—177).—The dry extract of milk previously treated with dichromate decreases appreciably from day to day, though much more slowly than that of untreated milk. The lactic acid formed slowly reduces the dichromate, so that the determination of the lactic acid content does not give a reliable correction for the value of the dry extract; some of the lactic acid is also converted by heat into dilactic acid which remains in the extract.

C. P. STEWART.

Milk powders as food. II. Existence of vitamin-E. L. T. ANDEREGG and V. E. NELSON (Ind. Eng. Chem., 1926, **18**, 620—622).—Skimmed milk powder, compared with whole milk powder, appears to be deficient in the proteins and vitamins necessary for growth and reproduction of rats and rearing of young; the deficiency cannot be remedied by the addition of butter-fat to the skimmed milk to make the chemical composition similar to that of whole milk. Cod liver oil incorporated with the skimmed milk gives poor results, as decomposition occurs, similar decomposition being observed with cod liver oil and other highly desiccated materials. Ethyl alcohol, wheat oil, or water, however, exert a protective influence. The addition of water to the skimmed milk powder, and administration of cod-liver oil separately, produced very good results, young of the fourth generation being reared successfully. These results are at variance with views previously expressed regarding the existence of vitamin-E (cf. B., 1925, 520). B. W. CLARKE.

Chemistry of flesh foods. No. 6. Canned meats. A. M. WRIGHT and (Miss) J. F. BEVIS (N.Z. J. Sci. Tech., 1926, **8**, 163—167).—A concentration of the food value occurs during the canning of meats, *e.g.*, tongues, mutton, etc., owing to evaporation of water during the parboiling of the meat previous to the canning process. Detailed analyses of fresh and canned meats are given.

B. W. CLARKE.

Feder number in relation to the examination of meat. H. W. DE KRUIJFF and G. L. VOERMAN (Chem. Weekblad, 1926, **23**, 296—299).—The Feder number, *i.e.*, the ratio of moisture to organic material other than fat, has been determined for a great number of different meats, and it is confirmed that the value should always be less than 4. For samples containing flour, *e.g.*, sausage meat, the ratio should be calculated on the content of organic matter not fat, less the flour content, in testing for addition of water. Addition of casein or milk powder may be detected by examining for lactose and calcium compounds. S. I. LEVY.

Examination of Canadian sprayed apples for arsenic. F. T. SHUTT (Analyst, 1926, **51**, 291—293.—Eight samples (each consisting of 6—12 apples) of apples from Nova Scotia, 8 from Quebec, 17 from Ontario, and 10 from irrigated orchards of British Columbia were examined for arsenic in the skin, calyx, and stalk respectively, and it was found that approximately half the samples were free from arsenic, a sixth showed traces less than 1/10,000 of a grain per lb., and a third contained quantities from 1/10,000 to 1/190 of a grain per lb.

D. G. HEWER.

Preparation and colloidal properties of pectin. (Miss) M. A. GRIGGS and (Miss) R. JOHNSTIN (Ind. Eng. Chem., 1926, **18**, 623—625).—Pure pectin is prepared from lemon albedo by first extracting with alcohol and subsequent extraction of the residue with 0·01*N*-hydrochloric acid at 90°. The pectin sol is freed from electrolytes by dialysis, the pectin precipitated by alcohol and flocculated in an electric field. The colloidal properties and conditions of gelation of pure pectin sols and of alcohol–pectin and sugar–acid–pectin sols are described.

B. W. CLARKE.

Food value of different grades of barley as produced by a modern seed purification plant. F. HONCAMP and W. SCHRAMM (Landw. Vers.-Stat., 1926, **104**, 285—296).—Digestibility trials with sheep indicate that the digestible protein and starch equivalent of various grades of barley, separated by the Schule system of seed purification, differ little in amount from those of the original grain or from one another. It is suggested, therefore, that all the first-class seed should be reserved for sowing and not used for feeding. C. T. GIMINGHAM.

Composition and digestibility of barley and its milling offals. F. HONCAMP and W. SCHRAMM (Landw. Vers.-Stat., 1926, **104**, 297—312).—The results of microscopical examinations of barley meals and various milling products of barley, and of digestibility trials with the same materials are recorded. A coarse barley meal showed the highest starch equivalent. C. T. GIMINGHAM.

Influence of degree of maturity on the composition of peas. C. F. Muttelet (Ann. Falsif., 1926, **19**, 283—290; cf. B., 1925, 186).—Ratios previously found to increase with the degree of maturity in the case of the American Marvel pea have now been worked out for the three varieties Caractacus, Serpette, and Express, and a general agreement with former results obtained. It is thus possible from determinations of starch, sugars, soluble and insoluble nitrogen, and cellulose to obtain an indication of the age of the product. D. G. Hewer.

Preserved peas in relation to their diameter. E. Lasausse (Ann. Falsif., 1926, **19**, 290—293; cf. B., 1926. 382).—A criticism of Muttelet's experiments and conclusions. D. G. Hewer.

Determination of sodium; applications. [Determination of sodium in mineral waters and milk.] L. Barthe and E. Dufilho (Compt. rend., 1926, **182**, 1470—1473).—Sodium may be determined by precipitation as the triple acetate of uranium, magnesium, and sodium (cf. Blanchetière, A., 1923, ii, 579) in the absence of organic matter and phosphates. If organic matter is present it should be destroyed by the nitro-sulphuric acid method without addition of permanganate. Phosphates are best precipitated by a uranium salt using hot solutions. This direct determination of sodium when used for mineral waters gives results differing from those obtained by the usual indirect methods, indicating that the generally accepted figures may be wrong. Thus for a Vichy water the direct and indirect methods gave 1·4064 and 1·5285 g. of sodium per litre respectively. The application of the method to the determination of sodium in milk is described in detail. Normal untreated milk contains from 0·345 to 0·484 g. of sodium per litre, and this method is very suitable for the detection of the addition of sodium bicarbonate to milk. W. Hume-Rothery.

Determination of free acid in silage. F. Mach and W. Lepper (Landw. Vers.-Stat., 1926, **104**, 317—320).—The use of phenolphthalein as an indicator for the determination of the total acidity in silage and similar acid food products gives results which are too high. The drop method with a specially sensitive litmus paper is recommended as giving satisfactory results. C. T. Gimingham.

Determination of crude fibre in the presence of products of animal origin. F. Mach and W. Lepper (Landw. Vers.-Stat., 1926, **104**, 313—316).—In the determination of crude fibre in such materials as fish and meat meals, hair, feathers, etc. a determination of the nitrogen in the fibre is essential. If asbestos is used for filtering, the change in weight on ignition should be determined in a blank experiment and allowed for. The amount of 1·25% hydrochloric acid used for the digestion should be increased from 200 c.c. to 400 c.c. if the material contains bonemeal. C. T. Gimingham.

Patents.

Artificial drying of crops. B. J. Owen (E.P. 251,577, 2.4.25. Addn. to 235,273; B., 1925, 648).—In addition to the central hollow space for the distribution of the heated air, one or more smaller auxiliary spaces are provided which ensure the more rapid and uniform drying of the material. G. W. Robinson.

Process for making a kola preparation. W. J. Wulff (E.P. 252,765, 24.11.25).—Raw kola nuts, as distinct from the fresh nuts, are subjected to a preliminary fermentation, which prevents drying or hardening, and are then cut up, soaked in water, fermented in their own juice, dried, roasted, and ground. Alternatively, the raw nuts are steeped in water before fermentation. The product is free from the bitter taste of the natural nut and the free caffeine content is very low. E. H. Sharples.

Drying hay and like crops. W. Donald (E.P. 252,533, 8.5.25).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Basic [dissociation] constant of morphine and its application in the titration of morphine. C. Morton (Pharm. J., 1926, **116**, 567—570, 593—597).—The hydrolysis constant of morphine hydrochloride at 30° (determined by E.M.F. measurements) is $1·914\times10^{-6}$, whence the dissociation constant of morphine is $6·27\times10^{-9}$. On theoretical grounds, the accepted methods of titrating morphine cannot be expected to yield accurate results, but by the back titration of acid solutions, using litmus, cochineal, and methyl-orange as indicators, fairly satisfactory results are obtained, owing to partial compensation of errors. Bromophenol-blue gives satisfactory results as indicator, and for the analysis of fairly pure morphine its use in combination with potassium hydrogen phthalate solution as colorimetric standard is recommended. A system of hydrogen and calomel electrodes devised for the E.M.F. measurements is described. B. Fullman.

Yocca, a new caffeine drug. E. Perrot and A. Rouhier (Compt. rend., 1926, **182**, 1494—1496).—From the bark and stems of the *yocca* plant of southern Colombia a crystalline alkaloid, agreeing in composition and properties with caffeine, is readily extracted. B. W. Anderson.

Determination of nicotine in tobacco. R. R. T. Young (N.Z. J. Sci. Tech., 1926, 8, 129—142).—Keller's method for the determination of nicotine in tobacco (cf. A., 1899, ii., 194) yields accurate results when slightly modified. Three successive extractions for ½ hr. with an ether-petrol mixture containing only 10% of ether are more satisfactory than one extraction for 3 hrs. with a 50% ether-petrol mixture, whilst cochineal or rosolic acid is to be preferred to iodoeosin as indicator. It is unnecessary to pass a current of air through the solution to expel ammonia, since experiments show that no ammonia is liberated by this method. B. W. Clarke.

Value of the D.M. index from the point of view of the toxicity of the arsenobenzenes. De Myttenaere (J. Pharm. Chim., 1926, [viii], 3, 497—506). A Valeur and L. Launoy (*Ibid.*; 506—507). Polemical. B. Fullman.

Analysis of glacial acetic acid. H. D. Richmond and E. H. England (Analyst, 1926, 51, 283—287).—A table is given showing the relationship between *d*, f.p., and percentage composition of glacial acetic acid, as all likely impurities of the acid affect these two physical properties. Propionic acid depresses the value of *d* on the average 0·0065 for each 1%, and the f.p. 0·485°. The percentage present may be calculated by dividing the difference between the value of *d* calculated as equivalent to the f.p. found, and that actually found, by 0·00135. Values of *d* calculated for different values of f.p. at intervals of 0·1° are given in a table. Aldehydes and ketones may be present in too small quantities to affect the *d* or f.p., but may be detected by Schiff's reagent or Denigès' mercuric chloride test respectively. D. G. Hewer.

Analysis of acetic anhydride. H. D. Richmond and J. A. Eggleston (Analyst, 1926, 51, 281—283).—The rise of temperature when 2 c.c. of acetic anhydride are added to 200 c.c. of a mixture of 94% of toluene (diluent) and 6% of aniline, in a vacuum flask, serves as a measure of the percentage of anhydride in the sample. A small and constant rise of temperature is given by acetic acid, but this is practically negligible for acetic anhydride of 90% and over. Trustworthy results may be obtained by multiplying the rise of temperature by a factor and ignoring the small corrections, which, however, are given in a table. Cooling corrections and heat capacity of the apparatus were not determined so that at present a factor must be worked out for each apparatus. The maximum divergence between 2 experiments was 0·037°. D. G. Hewer.

Anæsthetic gases. W. E. Brown and V. E. Henderson (J. Pharm. Exp. Ther., 1926, 27, 1—8).—Propylene is a better anæsthetic than either butylene or propane, since it does not stimulate the spinal centres, whilst the low concentration required allows the administration of sufficient oxygen to maintain a practically normal metabolism. H. P. Marks.

Camphor researches at Naples. A. De Dominicis and C. La Rotonda (Annali Chim. Appl., 1926, 16, 7—20).—Some results of experimental steam distillation of camphor (*Laurus camphora*) plants, 6—7 years old, from the Naples Botanic Gardens are given. The younger leaves yielded 0·38% of camphor and 0·19% of essential oil in May, and 1·12% of centrifuged camphor and 0·10% of oil in July. The maximum yield of camphor (1·14%) from full-grown leaves was obtained in May and July. The highest yield of oil was obtained from leaves gathered in September. No camphor and only traces of oil were obtained from the stems of the plants. The roots gave yields of oil ranging from 0·45 to 1·67%, according to the season, but no camphor. Dry leaves yielded 2·37% of camphor and 0·31% of oil. R. Sansone.

Essential oil of *Laurus nobilis*, L. V. Morani (Annali Chim. Appl., 1926, 16, 21—28).—A study of the essential oil of Italian laurel (*Laurus nobilis*, L.). 20 kg. of leaves on distillation with steam, yielded 256 g. of a canary-yellow oil, having a characteristic smell recalling that of eucalyptus oil. The oil had d_{15}^{15} 0·92073, α_D^{24} −18·35°, n_D^{18} 1·4712, solubility at 20° in 70% alcohol 1 : 10·5 (particles in suspension); in 80% alcohol 1 : 1·1 (clear); esters as $C_{10}H_{17}{\cdot}OAc$ 13·02%, total alcohols ($C_{10}H_{18}O$) 23·19%, free alcohols 12·95%. Acetylation by the method of Boulez, as modified by Schimmel (cf. B., 1907, 221), gave total alcohols 27·76%, free alcohols 17·52%, indicating the presence of tertiary alcohols. The approximate composition of the oil is 45% of cineole, 18% of terpineol and geraniol, 13% of acetic esters, 0·55% of free eugenol, 1·1% of eugenyl acetate, 3% of methyleugenol, 12% of terpene hydrocarbons, principally β-pinene and phellandrene, and 3—4% of a sesquiterpene. The Italian oil differs from other laurel oils by containing β-pinene in the place of α-pinene and terpineol instead of linalool. R. Sansone.

Constituents of ichthyol oils. Scheibler and Rettig.—See III.

Patents.

Manufacture of halogenated alcohols. Farbenfabr. vorm. F. Bayer & Co., and H. Meerwein (E.P. 251,890, 11.6.25. Addn. to 253,584; B., 1925, 738).—The aluminium alkoxide or halogenated alkoxide, used according to the previous patent for converting a halogenated aldehyde dissolved in a primary alcohol into a halogenated alcohol, is replaced by aluminium alkoxide to which aluminium chloride has been added. The conversion of butylchloral into trichloro-*n*-butyl alcohol is described. B. Fullman.

Preparation of soluble salts of substituted phenylarsinic acids. R. W. E. Stickings, and May and Baker, Ltd. (E.P. 252,099, 1.7.25).—Soluble piperazine salts of amino- and acylamidophenylarsinic acids and the hydroxy-derivatives of these are prepared by direct combination of the components in solution in water or alcohol. Examples are given of the preparation of the salts of 3-amino-4-hydroxyphenylarsinic acid, 3-acetamido-4-hydroxy-phenylarsinic acids, 4-aminophenylarsinic acid, and 4-dihydroxypropylaminophenylarsinic acid. The salts have therapeutic application. B. Fullman.

Preparing pentaerythritol tetra-acetate. H. T. Clarke, Assr. to Eastman Kodak Co. (U.S.P. 1,583,658, 4.5.26. Appl., 1.8.24).—250 pts. of pentaerythritol are refluxed with 1000 pts. of glacial acetic acid and 1 pt. of concentrated sulphuric acid (as catalyst). The temperature of the vapour is maintained at 105° so as to allow water vapour only to escape. When water evolution has ceased acetic

acid is distilled from the ester by raising the temperature of the mixture to 118°. A calculated quantity of anhydrous sodium acetate is then added to neutralise the sulphuric acid, and the pentaerythritol tetraacetate is distilled from the residue of sodium sulphate at a pressure of 9 mm. at 186°. It is recrystallised from benzene. R. B. Clarke.

Production of amines including the substitution products thereof and nitriles. K. F. Schmidt (E.P. 252,460, 27.2.25). — See U.S.P. 1,564,631; B., 1926, 216.

Manufacture of hexamethylenetetramine. H. Wade. From S. Karpen & Bros. (E.P. 252,609, 23.10.25).—See U.S.P. 1,566,820; B., 1926, 217.

Manufacture of alkyl esters of formic acid. J. Y. Johnson. From Badische Anilin & Soda Fabrik (E.P. 252,848, 22.4.25).—See U.S.P. 1,572,698; B., 1926, 462.

Monoiodo-oxindole and process of making it. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of W. Schoeller and K. Schmidt (U.S.P. 1,587,866, 8.6.26. Appl., 24.11.25. Conv., 10.12.24). —See E.P. 244,444; B., 1926, 514.

Preparation of solutions of derivatives of di[hydr]oxydiaminoarsenobenzene. S. R. MacEwen (U.S.P. 1,588,252, 8.6.26. Appl., 7.12.23).—See E.P. 221,565; B., 1924, 966. An alkaline reagent may also be added.

N-Acyl derivatives of 5-amino-3-chloro-4-hydroxybenzene-1-arsonic acid. I.-G. Farbenind. A.-G., Assees. of L. Benda and W. Schmidt (U.S.P. 1,588,382, 8.6.26. Appl., 7.11.25. Conv., 10.3.24).—See E.P. 230,487; B., 1925, 942.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Relation between the colour sensitivity of emulsions of the same kind, but of differing silver iodide content. R. Jahr (Phot. Ind., 1926, 609).—A series of emulsions containing respectively 1, 2, 3, and 4 g. of potassium iodide per 100 g. of silver nitrate, and sensitised with Erythrosin, were prepared in the same way. Sensitisation by the dye takes place most readily when no iodide is present. With more than 2% of silver iodide sensitisation begins to decrease, whilst with 4% of silver iodide there is practically no sensitisation. W. Clark.

Nitro-compounds as desensitisers. Lüppo-Cramer (Phot. Ind., 1926, 629—630).—Contrary to the observations of other workers, it is found that nitrates have no influence on the sensitivity of photographic plates. A desensitising action of aqueous sodium nitrite cannot always be observed. The strong desensitising action of a colourless nitrite solution on the direct blackening of silver chloride-gelatin plates or precipitated silver chloride cannot be due to absorption of the blue part of the spectrum by the nitrite (cf. Perley, Eder's Jahrbuch, 1913, 446). The view that, in general, nitro-compounds are desensitisers is not confirmed. The NO-group in sodium nitroprusside is not responsible for the desensitising action of this salt. W. Clark.

Photochemical processes with silver halides. H. H. Schmidt (Phot. Ind., 1926, 608).—The absorption spectra of dyes adsorbed on silver halides and of the silver salts of the dyes agree with the sensitivity spectrum of the silver halide dyed with the particular dye. It is considered that this explains the discrepancy between the absorption spectrum of a dye and the spectral sensitivity it confers. W. Clark.

XXII.—EXPLOSIVES; MATCHES.

Chemical stability of [propulsive] explosives as an additive property. M. Tonegutti (Z. ges. Schiess- u. Sprengstoffw., 1926, 21, 81—84).—Lenze and Pleus (B., 1920, 468 A) and de Bruin (B., 1921, 903 A) have shown that the behaviour of nitrocellulose does not support the generally accepted view that the decomposition products of a small quantity of an unstable constituent cause a catalytic decomposition of the remainder of the explosive. The investigation has now been extended to nitroglycerin propulsive powders such as ballistite and cordite by using Taliani's method (B., 1921, 562 A) to measure the rate of decomposition of various batches of either explosive alone and when mixed in various proportions. It is found that the stability of a mixture of 2 ballistites or 2 cordites can be satisfactorily calculated, when the proportions and stabilities of the constituents are known, by assuming that the stability is an additive property. In mixtures of cordite and ballistite the stability is not additive, but remains close to that of cordite, the more stable constituent, the vaseline in the cordite appearing to retard the decomposition of the ballistite. S. Binning.

XXIII.—SANITATION; WATER PURIFICATION.

Determination of iodine in natural waters. Brubaker, Van Blarcom, and Walker.—See VII.

Patents.

Biological purification of effluents contaminated with phenols. H. Bach (G.P. 426,422, 11.12.24).—Effluent from gas-works etc. is purified, after a preliminary treatment such as dilution, if required, by mixing with sewage and running into tanks where compressed air is blown in from below, either continuously or discontinuously, to stir up the sludge and subject the effluent to oxidation and the action of the bacteria in the sludge. E. S. Kreis.

Biological purification of effluents contaminated with organic matter, and containing acids, or tending to become acid; *e.g.*, effluents from breweries, distilleries, etc. H. Bach (G.P. 426,765, 20.1.25).—The effluents are purified by adding sufficient sewage, leading into a tank filled with clinker etc., and blowing in air from the bottom. The purification is quick and efficient. E. S. Kreis.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUGUST 20, 1926.

I.—GENERAL; PLANT; MACHINERY.

Equipment for high-pressure reactions. F. A. Ernst (Ind. Eng. Chem., 1926, 18, 664—669).—The chief constructional materials used for making large-scale apparatus for carrying out reactions at high temperatures under pressure are chromium, nickel-chromium, and chromium-vanadium steels for work up to 500°, and nichrome containing 80—85% Ni and 20—15% Cr for higher temperatures. A steel containing 2·5% Cr, 0·2% V, and 0·37% C is suitable for ammonia converters working at 450—500°. The tensile strengths of the various high-temperature steels decrease slightly up to about 250°, then increase to a maximum at 300°, and finally decrease rapidly with further rise of temperature. Diagrams are given showing the construction of gas compression cylinders for laboratory work, hydraulic pumps, heads for pressure containers, high-pressure valves and connexions, high-pressure compressors, a magnetic regulating valve for high pressures, a relief valve, and an insulated high-pressure joint for an electric lead, together with details of their working and sphere of usefulness. The original must be consulted for full details.

A. R. Powell.

Measurement of surface temperatures. I. Portable thermocouple device compensated for heat losses. M. W. Boyer and J. Buss (Ind. Eng. Chem., 1926, 18, 728—729).—In order to compensate for heat losses when measuring the temperature of a surface up to 150°, the two wires of the thermocouple are each soldered to a 1-in. silver disc, on diametrically opposite sides, in such a way that the disc, acting as the hot junction, may be laid flat on the surface the temperature of which is to be measured. To prevent errors due to radiation an exactly similar couple is arranged just above the measuring couple, but insulated from it by a thin sheet of mica, and is heated on the outer side by means of a resistance grid to such a temperature that when both couples are connected to a galvanometer no deflexion occurs showing that both couples are at the same temperature, *i.e.*, the temperature of the surface. The whole device is mounted in a flat wooden block, 3 in. square and ½ in. thick, so as to be easily portable.

A. R. Powell.

See also A., July, 678, **Structure of precipitates** (Odén). 706, **Preparation of nickel membranes for ultrafiltration** (Manning). 707, **Apparatus for continuous percolation and for filtration in neutral atmospheres** (Evans); **Non-inflammable liquids for cryostats** (Kanolt).

Turbidity readings. Scott.—See XXIII.

Patents.

Regulation of evaporating, concentrating, and distilling apparatus. Soc. des Condenseurs Delas (E.P. 229,679, 18.2.25. Conv., 23.2.24).—In apparatus of the type described in E.P. 201,123 (B., 1923, 1114 a) an automatic feed regulator is provided, preferably worked by the differential expansion of two arms of a bent tube, the upper arm being filled with steam from the vapour space and the lower arm containing water condensed from the arm above, at a level varying in equilibrium with the varying level of the liquor in the evaporator.

B. M. Venables.

Vertical film type evaporators. Griscom-Russell Co., Assees. of L. E. Sebald (E.P. 232,925, 11.2.25. Conv., 25.4.24).—In a vertical evaporator of the falling film type, short lengths of smaller tubes extend into the upper parts of the heating tubes, the liquor being fed through the annular spaces and the vapour passing out through the interiors of the smaller tubes. The heating steam surrounds the outside of the heating tubes. A spiral vane may be placed in each annular space to impart a whirling motion to the falling film of liquor and the operation may be governed by maintaining a constant depth of feed liquor above the upper tube plate, surrounding the smaller tubes. The rate of evaporation may be regulated by controlling the rate of egress of concentrated liquor, most of which is returned by a circulating pump for re-evaporation. (Reference is directed under Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 154,355, 131,365, 28,537 of 1913, 22,670 of 1911, and 3006 of 1888.)

B. M. Venables.

Evaporating apparatus. Blair, Campbell & McLean, Ltd., and J. R. Phillips (E.P. 252,505, 4.4.25).—In an evaporator comprising a number of superposed effects, the combined liquor and vapour passes from any calandria to the corresponding separator through a passage surmounted by a dome-like deflector, which can be raised or lowered so as to vary the size of the annular opening to suit the density of the liquor and rate of evaporation.

B. M. Venables.

Apparatus for boiling ["bumping"] liquids. Siemens-Schuckertwerke Ges.m.b.H. (E.P. 235,192, 2.6.25. Conv., 3.6.24).—A device for concentrating liquids, *e.g.*, the sulphuric acid used in an absorption refrigerator, which have a tendency to boil unsteadily, comprises a tube surrounded by a source of heat. The liquid and vapour rise in the tube, which is so arranged that it is never

completely free from vapour. This may be effected by forming bulges in the tube or making it in a sinuous or helical form, so inclined that though the general slope is upwards some portions of the tube have a downward slope. B. M. Venables.

Pulverising mills. Raymond Bros. Impact Pulverizer Co., Assces. of J. Crites and W. H. Vogel (E.P. 248,357, 16.2.26. Conv., 2.3.25).—In a pulveriser comprising an impact chamber, and preferably also a fan chamber, the outlet from the pulverising chamber is regulated by means of a ring member with a conical opening which when rotated by external means also moves longitudinally by the agency of cam-shaped slots and alters the size of the exit opening. B. M. Venables.

Grinding discs for use in grinding mills. A. Blume (E.P. 251,778, 15.5.25).—In a grinding mill where two toothed discs rotate in opposite directions, the discs are arranged to have several concentric grinding zones with teeth that decrease in size from the centre outwards. Venting grooves, extending through all the zones, are arranged tangentially to a small circle at the centre of the disc, and the teeth between any two venting grooves are arranged parallel to that groove which is in the front of the teeth in the direction of rotation of the disc. B. M. Venables.

Crushing or grinding apparatus. W. Chambers (E.P. 253,055, 16.3.26).—The apparatus comprises a number of beaters or pestles rotating within a grinding ring or mortar which is supported on rollers so that it can rotate under the influence of the beaters. The frame carrying the rollers and ring, also the main shaft of the beaters, are spring-mounted and the arms of the beaters are made of resilient material. B. M. Venables.

Mixing, disintegrating, or homogenising machine. H. E. Austin, Assr. to Amer. Creosoting Co. (U.S.P. 1,587,063, 1.6.26. Appl., 18.5.25).—Two elements rotating relatively to one another are formed from segments of the surface of a paraboloid of revolution. The space between the two surfaces is not uniform and can be increased, the increase at the feed being proportionally greater than at the discharge end. In consequence discharge is hindered and economical grinding is effected. E. S. Kreis.

Disintegrating apparatus. J. E. Kennedy and L. A. Hunt (U.S.P. 1,587,653, 8.6.26. Appl., 26.3.25).—A revolving drum is divided into grinding and collecting chambers by means of a perforated partition, and screening of the material is effected partly by the partition and partly by means of screens situated in another chamber surrounding and concentric with the drum. The oversize from the inner compartment of the screening chamber passes through passages in the circumference of the drum back to the grinding chamber. The fine material from the outer compartments passes through a transverse passage situated in the collecting chamber to the axial outlet of the mill. B. M. Venables.

Pulverising machine. D. T. Fisher, Assr. to A. M. Read (U.S.P. 1,587,769, 8.6.26. Appl., 17.9.20. Renewed 19.10.25).—A revolving horizontal barrel is divided into separate grinding chambers by perforated partitions, each partition having, on the face further from the feed end, a number of vanes extending inwards from the circumference and adapted to propel material longitudinally away from the partition. B. M. Venables.

Removing suspended matter from gases. R. W. Bailey, and Metropolitan-Vickers Electrical Co., Ltd. (E.P. 251,682, 2.2.25).—Various forms of apparatus are described all of which embody the principle that collecting material, such as fibrous material, perforated metal, or metal wire fabric, is supported on both sides of easily removable walls, which alternate with fixed walls that serve only as guiding means for the gases. The cross-sectional area of the ducts between the fixed and removable walls preferably decreases gradually in the direction of flow of the gases, and arrangements may be made for cutting out any of the ducts while the collecting material is being cleaned. B. M. Venables.

Separating solid particles from gases. Chance and Hunt, Ltd., and W. A. S. Calder (E.P. 252,271, 24.2.25).—The gas is passed through one or more orifices and impinges on a wall placed at a distance from the orifice equal to about one-quarter or one-half the diameter of the orifice. The gas preferably passes only once through an orifice, and a liquid or vapour may previously be added to it to aid the deposition of the solid particles. The wall may take the form of a rotating disc, which may be provided with scrapers for removing deposited matter both from the interior of the orifice and the space between the wall and the orifice. B. M. Venables.

Separating solid and liquid constituents from gases and vapours. H. Szamatolski and A. Blohm (E.P. 229,305, 11.2.25. Conv., 13.2.24).—A number of sheet metal elements with stamped out tongues or baffles are assembled as shown in the figures to form a continuous separating zone, *a*, through which the gases are passed, and a number of trap chambers, *b*, in which the material separated by impact against the baffles is collected.

FIG. 4.

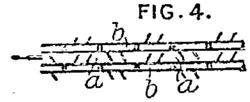
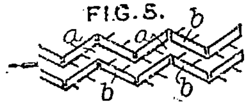


FIG. 5.



B. M. Venables.

Pyrometer sheath. W. C. Johnson (E.P. 251,750, 4.4.25).—A sheet of chromium steel is rolled to form a tube with the seam left open, say, 1/32 in., the seam is then electrically welded using a pencil of substantially the same material, the metal being allowed to flow through the seam to form a ridge both inside and outside the tube; a metallic mandrel with a longitudinal groove

coinciding with the seam may be used to produce a chill on the metal of the weld. The proportion of chromium in the steel should be at least 15%, and the welding voltage should be between 33 and 35 volts. The end of the tube is preferably formed by forging down to a small diameter and closing the resulting small end by welding. Chromium-steel tubes made in this way have withstood satisfactorily for extended periods a furnace temperature of 1260°.

B. M. Venables.

Centrifugal liquid purifiers. De Laval Chadburn Co., Ltd., and W. R. Chadburn (E.P. 252,421, 27.1, 3.2, 5.2, 9.2, 16.2, and 25.2.25).—The liquid, *e.g.*, a lubricating, transformer, or vegetable oil, or boiler feed-water, to be freed from absorbed oxygen, or beer or wine to be purified in presence of carbon dioxide, is sprayed from the bowl of the centrifuge in direct contact with inert or otherwise desirable gas delivered from a gas container into the stationary casing through a valved pipe connexion. Air may be preliminarily removed from the apparatus by a vacuum pump, and the gas-supply valve is then preferably regulated to maintain the pressure in the system above atmospheric. When economical use of the gas is necessary, the liquid is delivered into a closed collecting vessel from which the gas is re-circulated through the casing. The liquid is withdrawn from the collecting vessel while maintaining a constant liquid level therein to prevent escape of the gas. Separation of the gas from the liquid may be facilitated by the provision of partitions, inclined to the vertical, within the collecting vessel. When different liquids, *e.g.*, transformer oil and water, are separated into different compartments within the casing, each compartment is connected to a separate collecting vessel, and the two vessels are connected to equalise the pressure.

H. Holmes.

Apparatus for separating liquids from solids. H. C. Behr (U.S.P. 1,586,395, 25.5.26. Appl., 24.7.22).—A foraminous mantle surrounds a foraminous conveyer and the two are rotated at different speeds so as to effect a continuous separation of a mixture fed at a predetermined rate into the space between them by a rotary pump. The feed pump is driven through worm and belt gears from the centrifugal mechanism. H. Holmes.

Storing and shipping containers for acids and alkaline liquids. P. Pick (E.P. 252,772, 2.3.25).—Closed inner and outer shells of ebonite or like material are provided with a completely enclosed reinforcing skeleton of perforated metal. The method of making the containers is described in detail. H. Holmes.

Drying materials [*e.g.*, pulverised fuel]. O. Y. Imray. From Internat. Combustion Engineering Corp. (E.P. 252,835, 27.3.25).—The damp material (before pulverising) is fed into a stream of hot gases and the mixture immediately passed through a cyclone separator. Part of the used gases from the cyclone may be drawn back by the circulating fan and used again, only sufficient fresh hot gases being drawn in (and a corresponding amount of used gases exhausted) to maintain the necessary conditions. The hot gases may be drawn from several sources at different temperatures, and means are provided so that if the material happens to arrive sufficiently dry it may pass directly to the pulveriser without passing through the drying cyclone apparatus. The rate of feed of material is governed by the rate of pulverisation.

B. M. Venables.

Apparatus for drying material. G. A. Galloway, Assr. to B. F. Sturtevant Co. (U.S.P. 1,587,582, 8.6.26. Appl., 11.9.25).—A number of courses of ducts shaped as inverted troughs are arranged at right angles to one another, the ducts extending in one direction being used as supply ducts and those in the other direction as collecting ducts. The supply ducts are fed with air from an external source or through external passages from the collecting ducts next below, and the collecting ducts receive air from the supply ducts below through their open bottoms. The material to be dried passes downwards by gravity over the ducts.

B. M. Venables.

Dehydrating plant. G. W. Booksh, jun. (U.S.P. 1,588,298, 8.6.26. Appl., 13.1.26).—The apparatus comprises a casing with interior walls so arranged that hot air supplied to a central space passes through lateral drying chambers to flues at the sides, then up and inwards to an outlet at the top. B. M. Venables.

Absorption towers. N. Wade. From Koppers Co. (E.P. 253,057, 25.3.25).—A device for the top of an absorption tower to catch any spray that would strike the walls and run down them, consists of a number of concentric vertical rings, stepped downwards with decreasing diameter and with spaces left between the rings. B. M. Venables.

Lixiviation or extraction of solid substances by means of volatile solvents. P. C. Rushen. From F. Krupp Grusonwerk A.-G. (E.P. 253,333, 11.5.25).—The volatile solvent moves in a continuous cycle through a collecting tank, lixiviation vessel, separator for coarse solids, filter for removal of slime, still for recovery of dissolved matter, condenser, and collecting tank. The temperature of condensation is adjusted so that the solvent is recovered hot enough to be used for lixiviation without additional heating. The solid matter moves counter-current to the solvent in the lixiviating vessel, which consists of a revolving conical drum provided with transverse baffles and lifting blades around the circumference, a continuous feed and discharge being maintained through hollow trunnions. The lixiviated solid matter collected in the separator and filter is dried by evaporation and the vapour returned to the solvent circuit. B. M. Venables.

Apparatus for the continuous treatment of solids with liquids. [Recovery of sulphur from

spent gas-purifying material.] K. Pfisterer (G.P. 426,385, 17.1.25).—Solid material such as spent gas-purifying material is charged into a mixing chamber surrounding the liquid jet in apparatus similar to that described in G.P. 341,829 and 398,640 (cf. B., 1924, 899), whereby intimate contact between the solid and the solvent is effected as they pass together through the apparatus. L. A. Coles.

Granulating mill. F. L. Bryant (U.S.P. 1,586,282, 25.5.26. Appl., 7.2.23).—Within a cylindrical casing which increases in diameter downwards in a number of steps, rotate a corresponding number of horizontal discs, the spaces between the discs and between the rims of the discs and the casing being so adjusted that granulation takes place without substantial attrition or heating.
B. M. Venables.

Heating method and apparatus. F. X. Govers (U.S.P. 1,586,987, 1.6.26. Appl., 2.4.21).—For endothermic and exothermic reactions taking place in the neighbourhood of, say, 400°, a temperature-controlling medium is used which is a solid in the neighbourhood of 50° and a stable liquid at the temperature in question, as for instance a lead–tin alloy. This medium is brought into thermal contact with the charge and is then circulated to the furnace or cooler as the case may be. E. S. Kreis.

Heat treatment of chemical compounds. F. X. Govers (U.S.P. 1,587,115, 1.6.26. Appl., 8.8.23).—In a cylindrical heating vessel provided with an axial stirrer with radial arms, the arms are provided with vanes so arranged that the liquid is directed radially against the walls of the vessel as a result of the revolution of the stirrer, with the consequence that fresh liquid is always coming into contact with the heating surface and also that deposit on the heating walls is lessened. E. S. Kreis.

Regeneration of decolorising materials. L. Gurwitsch (G.P. 427,805, 14.10.24).—Spent decolorising material, such as fuller's earth, charcoal, alumina, or silica gel, is regenerated by extraction with mixtures of benzol or benzine with alcohols or ketones. L. A. Coles.

Method for making solid substances crystallise out of their solutions. Soc. des Condenseurs Delas (E.P. 238,215, 24.7.25. Conv., 6.8.24).—See G.P. 412,968; B., 1925, 699.

Grinding mill. K. Middleboe, Assr. to F. L. Smidth & Co. (U.S.P. 1,589,302, 15.6.26. Appl., 6.8.25. Conv., 21.8.24).—See E.P. 238,866; B., 1925, 868.

Production of metallic catalysts. E. J. Lush, Assr. to Technical Research Works, Ltd. (U.S.P. 1,590,897, 29.6.26. Appl., 11.5.25. Conv., 16.7.24).—See E.P. 241,278; B., 1925, 990.

[Analytical] crucibles. W. & J. George, Ltd., and J. D. M. Smith (E.P. 252,901, 5.6.25).—See J.S.C.I., 1925, 539 T; A., 1926, 41.

Air or gas filters. A. B. Cleworth & Co., Ltd., and A. B. Cleworth (E.P. 253,606, 19.3.25).

Machines for grinding, refining and mixing cocoa beans, chocolate, chemicals, and such like. J. McIntyre (E.P. 253,835, 27.2.26).

[Temperature-sensitive devices for] controlling chemical reactions. Brit. Thomson-Houston Co., Ltd., H. W. H. Warren, R. Newbound, and L. M. T. Bell (E.P. 253,984, 20.3.25).

Atomising liquids containing solid matter in solution or in suspension. P. Muller (E.P. 254,020, 27.3.25).

Apparatus for cooling liquids. C. Dunkelberg (E.P. 254,073, 26.5.25).

Electrodes for use in electrolytic systems for protecting boilers and the like. A. Kirkaldy (E.P. 254,188, 14.12.25).

Drying apparatus (E.P. 252,858).—See II.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Resins in coal. Composition of coal. W. Francis and R. V. Wheeler (J.C.S., 1926, 1410—1412).—By the oxidation of the vitrain portion of bituminous coal with air or hydrogen peroxide, followed by extraction with alkali, whereby the ulmin constituents are removed, pale yellow rods about 0·02 in. long and 0·002 in. diameter are obtained. When a polished and etched surface of vitrain is examined microscopically a cellular structure is observed, and the rods of resin can be seen in the positions they would be expected to occupy in the original structures. C. J. Still.

Origin of pyrites in coals. E. Donath and F. Vykypiël (Brennstoff-Chem., 1926, 7, 153—155).—Iron sulphides are produced if organic substances (*e.g.*, wood, albumin) are impregnated with solutions of ferrous or ferric sulphate and heated in an air oven to 200—300°, the organic material becoming charred. The nature of the disulphides found in coals furnishes a clue as to whether they were produced during or after the formation of the coal, the presence of pyrites and marcasite, or of pyrites alone, indicating sulphide production at an elevated temperature during coal formation.
W. T. K. Braunholtz.

Coal blending. D. Brownlie (Iron and Steel Inst., May, 1926. Advance proof, 42 pp.).—A review of the general principles of coal blending, as applied particularly to both high- and low-temperature carbonisation. Coal blending is often of advantage for boiler firing with ordinary or pulverised fuel and for preventing the caking of charges in gas producers. Coals which contain exactly the right amount of resinous material to give a homogeneous, coherent coke or semi-coke without swelling are rare, and in general it is advantageous to resort to

carefully controlled blending of strongly swelling bituminous coals, having an excess of resinous material, with non-swelling and non-caking coals, or other fuel deficient in this respect, *e.g.*, high-temperature coke breeze, low-temperature semi-coke (prepared from the same coal as that with which it is blended, or from any other suitable coal), oxidised or preheated coals in which the swelling properties are destroyed. High-grade coke may also be produced by carbonising a mixture of anthracite or other non-caking coal with tar, pitch, oils, or petroleum and other natural products. The principle of blending has found little application, however, in the brown coal, lignite, and shale carbonisation industries; nor have the possibilities of "chemical blending" been fully explored, *i.e.*, blending of coals with fuel products that have first been treated chemically (*e.g.*, coal bitumen) or with materials (*e.g.*, soap emulsions, barium carbonate) which have an advantageous influence on the resulting coke. The review, which contains photographic illustrations, concludes with a brief reference to a number of typical low-temperature carbonisation processes, in which fuel blending plays an important part. W. T. K. Braunholtz.

Correction for radiation in calorimetric determinations of calorific value. L. Niederstrasser (Brennstoff-Chem., 1926, 7, 185—186).—Langbein's simplified form of the Regnault–Pfaundler correction (J. pr. Chem., 1889, 39) is recommended provided the rise in temperature in the main determination is most rapid at the beginning and that the temperature of the calorimeter water is only so far below that of the water jacket that a minimum heat interchange takes place in the preliminary experiment.
W. T. K. Braunholtz.

Gas yield of various solid fuels as a criterion of their combustibility and behaviour in the fire. M. Dolch (Brennstoff-Chem., 1926, 7, 133—139).—Not only the content of volatile matter in a fuel, but also its gas yield and the composition and calorific value of the gas should be considered in evaluating the fuel. In the case of coals, the ratio (expressed as percentage) of the heat available from the gas alone to the total heat available from the fuel is in fairly close agreement with the gas yield (expressed as percentage by weight of the fuel), but in the case of lignite, semi-coke, or coke this agreement does not hold good, so that the evaluation of the latter on the basis of their content of volatile matter or gas yield is fallacious. The calorific values of the gases yielded by a variety of solid fuels are tabulated, together with the percentage of the total heat contributed in each case by the hydrogen, carbon monoxide, and methane present. W. T. K. Braunholtz.

Coke testing. W. A. Haven (Amer. Iron and Steel Inst., May, 1926; Engineering, 1926, 172, 13).—The relative rate of combustion of coke for blast-furnace purposes was determined by introducing 1 g. of carefully sized coke in a platinum boat into a combustion tube maintained at 980°, and determining the loss in weight at 1 min. intervals. Average results expressed in cg. per min. were taken as the burning speed. High ash content was a detriment to rapid combustion and cellular structure influenced the combustibility. The factors involved are apparently developed from the original coal and variation in the method of coking is of little importance. A modified tumbler test was used also to determine the character of the coke, on the assumption that the portion which withstood disintegration would represent the portion most likely to reach the region of the tuyères of the blast furnace. This test showed advantages in coke made from coal with a low content of volatile matter. C. A. King.

Low-temperature carbonisation. D. Brownlie (Proc. S. Wales Inst. Eng., 1926, 42, 273—376).—A detailed historical and general review of processes for producing free-burning, smokeless fuels, involving the mechanical compression or briquetting of the charge before or during carbonisation, whereby bituminous coal, in the form of slack or dust, together with a considerable amount of material poor in volatile matter, can be converted into a high-grade product in large pieces. The free-burning property is due, in these fuels, to the very close-grained structure of the carbonised material, irrespective of its content of volatile matter or the temperature of carbonisation. As examples of continuous coking processes in which the viscous charge is compressed during carbonisation, details are given of the Summers process, in which a hydraulic ram is used, and of the three adaptations of Fischer's principle, in which compression is effected by the combined influence of centrifugal force and a heavy loose roller, viz., the Dobbelstein, Raffloer, and Meguin processes. A second general method, involving briquetting by means of pitch or other externally produced binder followed by carbonisation at low or high temperature, is exemplified by detailed accounts of the "carbocoal" process (high-temperature carbonisation of a briquette of semi-coke and pitch), the "Midland Coal Products" process (combined carbonisation and gasification of pitch briquettes of non-coking slacks, alone or blended with coking slacks), and the "Sun Fuel" process (carbonisation of briquettes containing pitch and a mixture of small anthracite and bituminous coal). The third general method, viz., briquetting without pitch or other externally produced binder, followed by carbonisation, includes the "Pure Coal Briquette" process (briquetting of blended fuels by means of high pressures, followed by carbonisation at a medium temperature), and the Delkeskamp process (briquetting a mixture of coal and a colloidal solution of the same coal or any other carbonaceous material, followed by carbonisation at low or high temperature). The paper contains numerous illustrations and statistical tables.
W. T. K. Braunholtz.

Determination of yield of low-temperature tar with the aluminium retort. E. Fleischmann (Brennstoff-Chem., 1926, 7, 186—187).—Tar and aqueous distillate are collected in a water-cooled, graduated and tared glass tube, which is re-weighed

and then filled with benzene, shaken, and centrifuged. The water separates out sharply and its volume is read off, the tar yield being determined by difference.
W. T. K. Braunholtz.

Oil from coal. H. Nielsen (Gas J., 1926, **174**, 591—592, 650—653, 732—734).—Processes of low-temperature carbonisation, the Bergius process, and Fischer's "Synthol" process are compared from the point of view of the economic conversion of coal into oil. Emphasis is laid on the importance of producing a "primary" oil by low-temperature carbonisation. The oil from the "L. & N." process (Nielsen and Laing, B., 1924, 122) satisfies Fischer's criteria for a primary oil (B., 1919, 941 A), whilst that from an externally heated horizontal retort at 560° fails to do so. If a true primary product is obtained the neutral oils from all bituminous coals are identical. The apparent discrepancy between this result and the statement of Sinnatt and King (J.S.C.I., 1925, **44**, 413 T) is due to the use by the latter of a retorting system yielding a mixture of primary and secondary products. Information about oils derived only from distillation curves may be misleading and should be supplemented by determinations of specific gravity. The necessary distillation conditions for obtaining a true primary oil are outlined. The principal essential condition consists in the rapid removal of the products of distillation from the retort by a current of inert gas. The "L. & N." neutral oil is apparently identical with Bone's "fraction 1" obtained on extracting coal with benzene under pressure (J.S.C.I., 1925, **44**, 291 T). The high-boiling fractions of the "L. & N." oil are characterised by great thermal stability, and form good lubricating oils. The results of Heyn and Dunkel on the berginisation of a Silesian coal (Brennstoff-Chem., 1926, **7**, 20, 81) are compared with those of the "L. & N." process applied to a similar coal. The percentage of lubricating oil in the Bergius product is negligible. The Bergius process appears to be essentially a "cracking" process. The Fischer and Tropsch "Synthol" process (B., 1924, 152, 1001) is discussed and it is concluded that the most efficient method at present available for the conversion of coal into oils consists in (*a*) low-temperature carbonisation in the "L. & N." retort, and (*b*) use of the coke as a source of water-gas and conversion of the latter into "Synthol."
A. B. Manning.

Decomposition of "Synthol" at atmospheric pressure. H. Tropsch (Abh. Kennt. Kohle, 1925, **7**, 49—50; Chem. Zentr., 1926, I., 3583).—"Synthol" (5·8 g.) on heating to 400—480° at atmospheric pressure yielded 3·9 g. of carbon, 0·3 g. of carbon dioxide, 0·7 g. of carbon monoxide, 0·5 g. of hydrogen, 0·2 g. of methane, and 0·1 g. of hydrocarbons, these products being different from those obtained by heating it under pressure in autoclaves (cf. B., 1923, 1009 A).
L. A. Coles.

Rational utilisation of Rumanian lignite. I. Distillation at low temperatures. N. Danaila and I. Blum (Bul. Chim., Soc., România Stiin., 1926, **28**, 35—75).—The results of laboratory and small working scale tests on Rumanian lignites are described. The coke obtained by carbonisation at 500° in a revolving retort was friable and burnt easily but did not smoke. It did not contain too much ash and had a higher calorific value than the original lignite. The tar amounted to 6·8—9% on the dry basis, and the gas 50—130 cub. m. per ton of 3335—9095 cals. per cub. m. The utilisation of the coke and tar are deciding factors in the commercial feasibility or otherwise of the process, and depend upon the friability of the coke and the phenol content of the oils respectively. Up to the present low-temperature carbonisation cannot be applied with success unless the coke from inferior lignite is gasified or used to make good-quality briquettes.
R. A. A. Taylor.

Gas-washing. G. Weissenberger (Gas- u. Wasserf., 1926, **69**, 493—496, 528—531, 549—554; cf. B., 1925, 388, 697).—Tetralin has theoretical advantages over creosote or lignite tar oil as a benzol wash-oil and has now been tested on the working scale at a coke-oven installation and at a small gasworks. Although tetralin has a lower boiling point than ordinary wash-oil, with a suitable debenzolising column a very good separation between benzol and tetralin is obtained. Details given show only about 2% of the latter in the crude benzol, which is of low gravity and low in unsaturated hydrocarbons. The used tetralin tests are similarly good. The use of tetralin in an unaltered gas-washing plant designed for wash-oil gives less striking results, but if the benzol produced is to be used as motor spirit a proportion of tetralin in it is unobjectionable. It is preferable, however, to use a tall washer of small cross-section and a restricted flow of the circulated liquid when working with tetralin. Such a washer works badly with creosote wash-oil. Tetralin is less rapidly affected by tarry impurities than creosote and owing to the considerable increase in the solubility of naphthalene in tetralin with rise of temperature it is a very suitable agent for removing naphthalene from gas. Although pure tetralin has *d* 0·9709 this figure rises little with addition of naphthalene etc. in solution and no difficulty in separation should occur if direct cooling is used. It appears to have no tendency to form emulsions with water. The loss of tetralin through vaporisation with the gas was found over a period of 1 month to average 1·3 g. per cub. m. of gas. It is claimed that the use of tetralin protects gas mains against corrosion and removes naphthalene deposits.
C. Irwin.

Extraction of oil shales and lignites with tetralin. E. Berl and W. Schmid (Brennstoff-Chem., 1926, **7**, 149—153, 181—185).—Tetralin under pressure (*e.g.*, 14 atm.) and at a temperature of 200—270° extracts more from oil shales and lignites than does benzene under similar conditions. The material extracted from a sample of Cassel lignite, when further fractionally extracted with various

solvents, yielded 11·2% of plastic waxes, 40·5% of resin, 20·8% of pitch, and 12·3% of acidic compounds (acids and phenols). W. T. K. BRAUNHOLTZ.

Chemical constitution of a fraction of Peruvian petroleum boiling between 150° and 350°. W. F. SEYER and J. L. HUGGETT (Trans. Roy. Soc. Canada, 1926, [iii], 20, III, 193—199).—A sample of crude oil from the Lobitos Oilfield in Peru had d_{20}^{20} 0·834, and contained 0·10% of sulphur; 75% distilled between 65° and 370°. The hydrocarbons $C_{10}H_{20}$, $C_{11}H_{22}$, $C_{12}H_{24}$, $C_{13}H_{26}$, $C_{16}H_{32}$, $C_{17}H_{34}$, $C_{18}H_{36}$, $C_{13}H_{24}$ $C_{14}H_{26}$, $C_{15}H_{28}$, and $C_{19}H_{36}$ were identified in the fraction investigated. The hydrocarbons containing 14 or more carbon atoms were optically active. The values of d^{20}_{20} and n_D^{20} of the hydrocarbons increased somewhat irregularly with increasing molecular weight from 0·788 to 0·854, and from 1·435 to 1·469, respectively. The molecular volumes point to a ring structure with side chains. Some evidence was also obtained of the presence of two lower members of the C_nH_{2n} series, C_8H_{16} and C_9H_{18}, having a pronounced odour of turpentine. The optical activity displayed by some of these hydrocarbons, as well as the terpene odour of the two unidentified oils, favours the plant-theory of the origin of petroleum. J. S. CARTER.

Relation between fuel deposition temperature and equilibrium boiling point. W. A. WHATMOUGH (Ind. Eng. Chem., 1926, 18, 609—612).—Determination of fog points of combustible mixtures in internal-combustion engines (*i.e.*, the temperature at which visible droplets of liquid form in a flowing fuel–air mixture) gave results indicating that the equilibrium b.p. of a motor fuel is a measure of its volatility, and the principal factor in its valuation. The temperature of the first 10—20 c.c. of Engler distillate indicates the degree of ease of starting. The equilibrium b.p. determines the necessary induction pipe and mixture temperatures. In the experiments the fuel was evaporated in a flash-type balanced pressure boiler of a capacity only sufficient for a few revolutions of the engine. Fog points were determined, and the relation of these to equilibrium b.p. temperatures is shown by table and graph. The linear relationship between fog points and equilibrium b.p. agrees with Barnard and Wilson's figures for light fuels (cf. B., 1925, 487), but not for heavy fuels. The 85% boiling point of an Engler distillation does not represent accurately the equilibrium b.p. of all mixed motor fuels. H. MOORE.

Life of fuller's earth used in the vapour-phase treatment of cracked distillates. M. R. MANDELBAUM and P. S. NISSON (Ind. Eng. Chem., 1926, 18, 564—566).—When clays used for decolorising oils by percolation are revivified by ignition their value becomes less with each re-burning, but the value of fuller's earth for vapour-phase refining remains undiminished after burning. The efficiency of fuller's earth in vapour-phase refining is practically the same for any cracked distillate. Better yields are obtained in large-scale working than in the laboratory. To determine the efficiency of the original and re-burned earths, colour stability was taken as a decisive test. Gumming tests were found to be uncertain and not to give reproducible results, and the methylene-blue test was erratic in its results with fuller's earth. The turpentine temperature reaction (cf. Venable, B., 1923, 424 A) is much affected by the presence of moisture. The clay was reburned in a gas-fired muffle furnace, being frequently stirred, and was found in some cases to be more efficient than the unused clay. Relatively low temperatures of ignition, not exceeding 550°, were found to give the best results. H. MOORE.

Method of measuring viscosity at higher temperatures. H. M. MERCER (J.S.C.I., 1926, 45, 203—205 T).—A new method suitable for oils, molten waxes, tars, etc., at temperatures above 100° in which a small quantity of the oil or other material at a definite temperature is drawn into a capillary and its weight determined is described in detail. Values obtained for representative oils and paraffin wax are represented graphically and a few values for pitch are given.

Naphthenic acids. XI. Naphthenic acids derived from petroleum before acid treatment. Y. TANAKA and S. NAGAI (J. Soc. Chem. Ind. Japan, 1926. 29, 115—121; J. Fac. Eng. Tokyo, 1926, 16, 183—188).—Kerosene distillate from Nishiyama crude petroleum before refining with acid was treated with caustic soda and a mixture of crude petroleum acids was obtained from the solution by acidification. This was distilled under 9 mm. pressure and the distillate converted into potassium soaps from which a mixture of crude naphthenic acids was obtained. When the crude naphthenic acids were distilled under 9 mm. pressure a considerable amount of phenolic compounds was obtained from the first fractions boiling below 150° The crude naphthenic acids were purified by means of their methyl esters. The naphthenic acids derived directly from the kerosene are practically indistinguishable from those obtained from the waste lye produced in the ordinary refining of the same kerosene (cf. B., 1926, 37). In the usual chemical treatment of petroleum, therefore, sulphuric acid does not act chemically on the naphthenic acids but on the phenolic compounds, most of which are removed. K. KASHIMA.

See also A., July, 668, **Heats of combustion of cyclic hydrocarbons** (KONOVALOV). 669, **Temperature of the acetylene flame** (HEINRICH). 673, **Adsorption of gases by activated charcoal at very low pressures** (ROWE). 689. **Explosive reactions in gaseous media** (GARNER); **Uniform movement during propagation of flame** (PAYMAN and WHEELER); **Ignition-point of gases** (DIXON, HARWOOD, and HIGGINS). 690, **Ionisation and gaseous explosions** (LIND); **Ignition of gases by sudden compression** (TIZARD and PYE). 693, **Radiation in gaseous explosions** (DAVID). 706 **Copper covers for calorimeter jackets** (WHITE).

Ammonium sulphate manufacture. HAILSTONE.—See VII.

Pyrogenic dehydration of fusel oil. GUIA and THUMIGER.—See XVIII.

PATENTS.

Apparatus for drying or dehydrating and distilling. W. DONALD (E.P. 252,858, 2.5.25).—Material to be dried or distilled is heated in a horizontal autoclave having end tube-plates, smoke-boxes at each end, and firing chambers between the smoke-boxes and the autoclave. Fire-tubes extend longitudinally through the autoclave from each firing chamber, passing through the opposed firing chamber, and terminating in the corresponding fire-box. Gas or oil is used as fuel. The material is supported inside the autoclave, out of contact with the fire-tubes, either on bogies or in the interior of a rotating perforated drum. In one form of the apparatus the autoclave and rotating drum are inclined, and means are provided for feeding the material into the upper end and discharging it from the lower end. Spiral worms inside the fire-tubes cause the products of combustion to take a tortuous path. The worms can be rotated, and so serve also to remove carbonaceous deposits from the tubes. In drying peat the pressure is allowed to rise initially to $2\frac{1}{4}$ atm. above atmospheric pressure, then partially released and kept at $\frac{3}{4}$ atm. above atmospheric pressure for half the remaining period of heating, drying finally being completed at atmospheric pressure. A. B. MANNING.

Retort for carbonisation of shale, lignite, coal, etc. E. L. SCHULTZ (E.P. 252,422, 27.1.25).—An annular vertical retort is heated from a central flue. Between the outer and inner walls of the retort is a casing provided with louvres arranged at a slight angle so that the material to be carbonised, entering from above, is fed in a layer against the inner heated wall of the retort. The casing with the louvres is moved up and down by means of an eccentric and so keeps the layer of material in motion. The gaseous products of carbonisation pass through apertures in the casing into the cooler outer space of the retort and thence to off-take pipes. The spent material is discharged intermittently at the lower end of the retort, discharge taking place automatically on the downward movement of the louvres.
A. B. MANNING.

Gas producers. H. T. WHITE, D. J. SMITH, and R. F. CLAYTON (E.P. 252,862, 5 and 7.7.25).—The producer consists of two concentric chambers, air passing up through the outer and down through the inner one. The producer-gas passes through a jacket surrounding these chambers and a cylindrical fuel hopper superposed on them. A second jacket is provided for preheating the air, which then, on its way to the producer, passes through and preheats the fuel in the hopper; and a third, outermost jacket is provided for further preheating the air by means of the engine exhaust gases. By-pass inlets for either air or steam or both may also be provided.
R. A. A. TAYLOR.

[Production of gas containing] hydrogen for the hydrogenation of naphthalene etc. E. A. PRUDHOMME (F.P. 603,639, 19.12.24).—Water-gas or a similar gas is passed over calcium hydroxide.
L. A. COLES.

Gas producer for the distillation of fine-grained or dusty fuel. SIEGENER MASCHINENBAU A.-G., and A. MENZEL (G.P. 427,038, 15.2.24).—The fuel passes downwards over a series of inclined plates heated by flues connected with the flue system in the walls of the furnace, and the issuing gases from the distillation are passed through this flue system, so as to utilise their heat for distilling the fuel without bringing them into direct contact with it.
A. R. POWELL.

Distillation of lignite and recovery of the volatile sulphur. F. SEIDENSCHNUR (G.P. 427,323, 31.8.20).—Lignite is treated in a continuously working retort with superheated steam and the issuing vapours are cooled to such a temperature that only the tar condenses. Part of the remaining gases is passed through a superheater and returned to the circuit, whilst the remainder is further cooled to remove most of the water, and sulphur or sulphur dioxide is recovered from the rest of the gas. The quantity of the steam used and its temperature are so regulated that a highly viscous tar containing a large proportion of undecomposed bitumen is obtained together with a gas sufficiently rich in sulphur to allow of its economic recovery and the return of sufficient steam and gas to the circuit without reducing the amount of sulphur recovered.
A. R. POWELL.

Washing or scrubbing coal or like gas. T. V. BLAKE (E.P. 252,867, 12.5.25).—A horizontal chamber is divided into sections by means of vertical hollow dividing plates and is partly filled with the scrubbing liquid. The gas passes out of one section through apertures in one side of the dividing wall just above the level of the liquid and into the next section through apertures on the other side of the hollow wall at the top. The scrubbing liquid is sprayed upwards from a pipe in the centre of the section just above the level of the liquid, and it is the film or spray so formed which scrubs the gas in its passage through the section. R. A. A. TAYLOR.

Gasification of [heavy] liquid fuels, for use in internal-combustion engines. A. V. DIAZ (E.P. 244,423, 24.7.25. Conv., 9.12.24).—The fuel passes through a vaporising coil and a vessel containing successive layers of coke and iron filings, heated by exhaust gases from the engine. Air is drawn through the coke by the suction of the engine, to maintain it at red heat and burn away deposited carbon.
L. A. COLES.

Production of light oils suitable for use as fuel for internal-combustion engines from [A] water-gas or similar gases, or [B] liquid and solid organic bodies. M. BRUTZKUS (E.P. 252,786–7, 5.3.25).—(A) Water-gas or similar gas, alone or mixed with hydrogen, is compressed in an apparatus resembling a Diesel engine, which may contain a catalyst, and, during the compression stroke,

the gas is cooled and the concentration of the initial gas is increased by forcing in more of it by means of a pump operating at a higher pressure than that inside the apparatus. The products pass into a cooler and liquefier, the residual gas being returned to the process. (B) Hydrocarbon oils are cracked and simultaneously hydrogenated by compression with hydrogen or gases containing it, *e.g.*, water-gas, in the presence of catalysts, in an apparatus resembling a Diesel engine, the pressure, temperature, and hydrogen concentration being varied in directions opposite to those provoked by the desired reaction. Polymerisation of the products is prevented by effecting the cracking process during the expansion stroke. Light oils can also be produced in a similar manner from sugar syrup or from finely-divided hay, wood, or coal (cf. E.P. 149,915, 155,776, and 217,747; B., 1922, 87 A, 736 A; 1924, 698).

L. A. Coles.

Purification of used lubricating oil. Aktiebolaget Separator (F.P. 602,304, 20.8.25).—The oil is heated and centrifuged to remove the heavier solid particles. A flocculating agent, such as trisodium phosphate, is then added to precipitate the colloidal materials present and the precipitate is removed by centrifuging. Finally, the oil is freed from volatile impurities by distillation.

A. R. Powell.

Emulsification of tar, bitumen, creosote, petroleum, heavy oils and the like. W. E. Billinghame (E.P. 252,449, 27.11.24).—Concentrated emulsions containing relatively small quantities of the material are used as emulsifying agents in the production of the desired emulsions. For example, an emulsion containing 1 pt. of neutral resin compound obtained by treating liquid resin with alkali, 1 pt. of tar, and 2 pts. of a solution of soluble casein or other suitable protein, is used for emulsifying 10 pts. of tar with 9 pts. of water. The emulsion may be thickened and stabilised by the subsequent addition of 10—50% of china clay. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 13,586 of 1912, 13,151 of 1905, and 14,391 of 1885.)

L. A. Coles.

Preparation of Trinidad pitch lake asphalt and the like. J. A. W. Pine (E.P. 252,802, 7.3.25).—The crude asphalt is pulverised, washed if necessary to remove salt and earthy material, compressed into blocks, and air-dried.

L. A. Coles.

Treatment of oils and hydrocarbons. [Production of blown oil asphalts.] J. S. Miller, jun., Assr. to Barber Asphalt Co. (U.S.P. 1,586,376, 25.5.26. Appl., 23.9.20).—Blown oil asphalts of any desired consistency are produced without distillation by spraying heated oil into a chamber in which it is brought in contact with a gaseous oxidising and condensing agent, the temperature and the proportions of oil and oxygen being regulated to prevent ignition and to minimise vaporisation of the oil.

L. A. Coles.

Rotary distillation retort. H. Nielsen (U.S.P. 1,589,417, 22.6.26. Appl., 15.2.21. Conv., 14.1.21).—See E.P. 178,537; B., 1922, 456 A.

Destructive distillation of wood. J. S. Morgan, Assr. to Thermal Industrial and Chemical (T.I.C.) Research Co., Ltd. (U.S.P. 1,590,901, 29.6.26. Appl., 1.10.21. Conv., 2.11.20).—See E.P. 176,438; B., 1922, 315 A.

Cleaning blast-furnace gases. F. Häring, Assr. to Freyn Engineering Co. (U.S.P. 1,589,803, 22.6.26. Appl., 26.8.21. Conv., 11.9.14).—See E.P. 17,081 of 1915; B., 1916, 958.

Burner apparatus for combustion of liquid, gaseous, or pulverised fuels. A. L. Mond. From E. H. Peabody (E.P. 253,684, 12.5.25).

[Gas] retort settings. Gibbons Bros., Ltd., and E. Masters (E.P. 253,778, 21.10.25).

Coke extractors for vertical retorts. F. J. and E. West, and West's Gas Improvement Co., Ltd. (E.P. 254,106, 30.6.25).

See also pages 649, **Centrifugal liquid purifiers** (E.P. 252,421); **Drying pulverised fuel** (E.P. 252,835); **Recovering sulphur from gas-purifying material** (G.P. 426,385). 650, **Regenerating decolorising materials** (G.P. 427,805). 664, **Phosphoric acid and generator gas** (E.P. 242,650 and 248,332).

III.—TAR AND TAR PRODUCTS.

Tar from low-temperature carbonisation of coal. E. Parrish and F. M. Rowe (J.S.C.I., 1926, 45, 99—106 T).—A low-temperature tar made under standard conditions was examined by extraction methods. The tar was obtained by the carbonisation of Pooley Hall coal at 600° in vertical retorts. It contained less than 1% of material insoluble in the usual organic solvents, except in the case of light petroleum, which precipitated 10·8% of a black pitch. By distillation, a number of pitches were obtained, which showed a gradation in their properties, according to the temperature to which distillation was carried. During the separation of the tar by reagents, small amounts of solids were precipitated, in particular on dilution of the oil soluble in alkali with ether. Small amounts of solid carboxylic acids were found. The phenols, consisting mainly of homologues of phenol, contained 28% of high-boiling material insoluble in light petroleum, which resembled the "rhetinols" found by Edwards (J.S.C.I., 1924, 143 T, 150 T). Like these, it was converted by oxidation into a powder similar to that previously separated from the phenols. Phenol itself was present to an amount of 0·7% of the tar, an abnormally high figure, not due, however, to overheating of the tar. Pyrocatechol was identified in small amounts. The bases were almost entirely secondary and tertiary compounds of high boiling point. The neutral oil contained 12·8% of naphthenes and paraffins, including 3·75% of solid paraffins. The neutral oil of b.p. up to 170° was much more

saturated in character than the higher fractions. Benzene was absent, but higher aromatic hydrocarbons were obtained as picrates, including naphthalene amounting to 0·1% of the tar. Sulphur compounds were present in all portions of the tar.

Measuring viscosity at higher temperatures. Mercer.—See II.

Patent.

Emulsification of tar etc. (E.P. 252,449).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Dyes from nitrosodialkylanilines, safranine, and Meldola Blue. A. Cobenzl (Chem.-Ztg., 1926, **50**, 494—495).—Whilst nitrosodimethylaniline forms a good crystalline hydrochloride, nitrosodiethylaniline and the safranines derived from it are best isolated as their double zinc salts. Heated in aqueous or acetic acid solution until the last trace of yellow colour has disappeared, nitrosodialkylanilines yield fast, water-soluble, grey cotton dyes, whilst by heating them with zinc oxide, zinc chloride, and aniline salts in dilute acid solution and oxidation with sodium dichromate, violet and blue safranines are obtained. *p*-Aminodiphenylamine directly oxidised with various aniline bases in aqueous solution gives economic yields (doubled if concentrated acetic acid solution is used) of rosanilines. If *p*-aminomethyldiphenylamine is used redder safranines are obtained, whilst the colour is bluer if *p*-aminotolyldiphenylamine is substituted. In the preparation of Meldola Blue, Naphthol Blue, etc., more satisfactory results and purer products are obtained if zinc chloride is added, or if the double zinc salt of the nitrosodialkylaniline is used directly, the dyes having the composition $(C_{14}H_{16}ON_2Cl)_2,ZnCl_2$ and $(C_{20}H_{20}ON_2Cl)_2,ZnCl_2$ respectively. Treatment of these with ammonia or aniline bases yields valuable blue dyes. The sulphonation of Naphthol Blue to make it suitable for dyeing wool may be effected either by direct sulphonation, or by using naphtholsulphonic acid in place of β-naphthol during the preparation. Details for the preparation of all these dyes are given. J. W. Baker.

Standardisation of Agalma Black 10 B. W. D. Appel, W. R. Brode, and I. M. Welch (Ind. Eng. Chem., 1926, **18**, 627—629).—The desirability of standardising commercial dyestuffs is emphasised, and three methods for estimating the strength of commercial Agalma Black 10 B are compared, namely titration with titanous chloride, spectrophotometric analysis, and comparative dyeings. A sample of the dye prepared in the laboratory and containing 99·2% of anhydrous dye was used as a standard. The spectrophotometric measurements were made in a Keuffel and Esser colour analyser and the negative logarithms of the spectral transmission of a 5 cm. layer of the dye solution at selected wave-lengths relative to that of the solvent in a similar cell plotted against the wave-length. The area between the curve thus obtained and the wave-length axis (within the limits λ 540—670) is proportional to the amount of dye in solution. The percentage purity was also calculated from the value of the curve at λ 620, the wave-length of maximum absorption; the average of the two results was taken as the spectrophotometric value. The concentration of the dye was about 3·3 mg. of pure dye per litre (on the basis of the titanous chloride titration in an aqueous buffer solution containing 0·01 g.-mol. each of sodium acetate and acetic acid; p_H 5). The comparative dyeing tests (0·4% of pure dye on white wool flock) were based on the results of the titanous chloride and spectrophotometric analyses, additiona dyeings being made where necessary; the relative strengths of the dyes were judged visually, a good commercial sample proving the most satisfactory standard. In the main the titanous chloride and spectrophotometric results agreed with the dyeing tests; in some cases where reducing substances other than the dye were present the titanous chloride figures were high; abnormal results obtained with a few commercial dyes appeared to be due to the presence of colouring matter other than true Agalma Black. In general, whilst neither titration with titanous chloride nor spectrophotometric analysis is sufficient alone, a combination of the two methods provides a fairly satisfactory means of evaluating Agalma Black 10 B, since where the two methods agree, the result is that obtained by the dyeing test. A standard of 60% purity is suggested. D. J. Norman.

Separation of the constituents of commercial xylidine. G. T. Morgan and W. J. Hickinbottom (J.S.C.I., 1926, 45, 221—222 t).—4-*o*-Xylidine forms a picrate sparingly soluble in alcohol, and this property has been utilised in devising a method for the separation of the amine from commercial mixtures of xylidines. The crude mixture is freed from *m*-xylidine by treatment with acetic acid; *p*-xylidine is removed as hydrochloride. The residual amines when treated in alcoholic solution with picric acid, insufficient in quantity to combine with all the bases present, deposit *4-amino-o-xylene picrate* in a crude state. Crystallisation from alcohol yields the pure picrate, from which pure 4-amino-*o*-xylene may be isolated by treatment with aqueous ammonia. From the residue 2-amino-*o*-xylene is separated by fractional crystallisation of the sulphates, and conversion of the more soluble fractions into acetyl compounds. Hydrolysis of the crude acetyl derivatives with hydrochloric acid liberates the isomeric amines, leaving unhydrolysed 2-acetamido-*m*-xylene in a pure state.

Synthesis of 2-*p*-aminophenyl-5-methylbenzthiazole etc.: isomerides of dehydrothio-*p*-toluidine and of Chloramine Yellow. Bogert and Allen.—See A., July, 743.

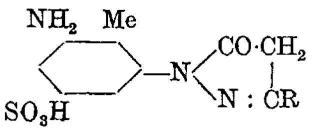
Patents.

Manufacture of yellow azo dyes. J. Y. Johnson. From Badische Anilin- & Soda-Fabr. (E.P. 252,617, 5.11.25).—Yellow azo *dyes* possessing excellent affinity for wool, silk, paper, and leather,

especially chrome-tanned leather, are obtained by nitrating the monoazo-dyes obtained from naphthylaminemonosulphonic acids and diphenylamine, or the *N*-nitroso-derivatives of such dyes. Thus the monoazo-dye from 22·3 pts. of a technical mixture of β-naphthylaminemonosulphonic acids and 16·9 pts. of diphenylamine is converted into the nitrosoamine with 8·5 pts. of sodium nitrite. The nitrosoamine is nitrated at 30—35° with 46 pts. of 65% nitric acid and 37 pts. of 98% sulphuric acid. The new dyes dye silk in a slightly acid boiled-off liquor bath or in a bath containing soap and Glauber's salt.

R. Brightman.

Manufacture of diazotisable azo-dyestuffs and intermediate products Chem. Works, formerly Sandoz, and M. Böniger (E.P. 252,957, 28.9.25).—By condensation of 6-nitro-2-tolylhydrazine-4-sulphonic acid with ethyl acetoacetate or ethyl oxaloacetate, followed by hydrolysis with hydrochloric acid and reduction with iron borings, new pyrazolone derivatives of the general formula,



where R=Me or CO_2H, are obtained. The symmetrical disazo dyes from a *p*-diamine and 2 mols. of these pyrazolones have no technical value. Valuable substantive dyes which can be diazotised on the fibre and developed, *e.g.*, with β-naphthol, *m*-diamines, or phenylmethylpyrazolone, giving shades varying from orange to red and dark-brown, fast to washing, and discharged by formaldehyde-hyposulphite, are, however, obtained when these pyrazolones are combined with the intermediate compound from 1 mol. of a tetrazotised diaryl-*p*-diamine and 1 mol. of an aryl-*o*-carboxylic acid; or alternatively, 1 mol. of the diaryl tetrazo-compound is combined with 1 mol. of the new pyrazolones and the intermediate compound produced is coupled with 1 mol. of an azo component. The direct dyeings can be made fast by after-treatment with *p*-nitrodiazobenzene or formaldehyde. Thus the intermediate compound from tetrazotised benzidine and *o*-cresotic acid yields with 5'-*sulpho*-3'-*amino*-2'-*methyl*-1-*phenyl*-3-*methyl*-5-*pyrazolone* (greyish powder, which reacts with 2 mols. of sodium nitrite, one mol, of diazo-compounds, and 3 equivalents of sodium hydroxide) a bright orange *dye*, developed on the fibre by diazotisation and coupling with *m*-tolylenediamine to a browner shade, with β-naphthol to bright reddish-orange shades. Similarly the intermediate compound from dianisidine and salicylic acid with 5'-*sulpho*-3'-*amino*-2'-*methyl*-1-*phenyl*-5-*pyrazolone*-3-*carboxylic acid* (grey powder, reacting with 2 mols. of nitrite, 1 mol. of diazo-compounds and 2 mols. of sodium hydroxide) yields a red *dye*, developed on the fibre to bluish-red shades by coupling with β-naphthol. The intermediate compound from tolidine and 5'-sulpho-3'-nitro-2'-methyl-1-phenyl-5-pyrazolone-3-carboxylic acid with 2-amino-8-naphthol-6-sulphonic acid yields a violet solution, reduced by sodium sulphide to a violet-brown *dye*, the nitro-disazo-dye being slightly yellower. Benzidine and 5'-sulpho-3'-amino-2'-methyl-1-phenyl-5-pyrazolone-3-carboxylic acid give an orange intermediate compound which couples with the monoazo-dye from *p*-phenylenediaminesulphonic acid and *m*-tolylenediamine to give a bright red-brown trisazo *dye*.

R. Brightman.

Manufacture of monoazo dyes. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 253,457, 29.6.25).—Monoazo *dyes* giving pure, even shades on wool are obtained by coupling diazotised aromatic amines or their substitution products with ω-aminoalkyl-β-naphthylamines or their derivatives (cf. U.S.P. 1,543,569 and E.P. 249,717; B., 1925, 752; 1926, 433). The new dyes give good dyeings on cellulose acetate, provided they contain only one sulphonic or carboxylic group, which must be present in the diazo-component. Thus the dye from 2 : 4-dinitroaniline-6-sulphonic acid and ω-aminoethyl-β naphthylamine on treatment with acetic anhydride at 65—75° gives a product dyeing acetate silk a blue shade and wool a greenish blue. 4-Nitroaniline-2-sulphonic acid and the *monoacetyl* derivative of ω-aminoethyl-β-naphthylamine, m.p. 101—102°, give a product dyeing acetate silk a red-violet and wool a violet shade. Similarly 4-nitroaniline-2-sulphonic acid with the acetyl derivative of ω-aminoethyl-2-naphthylamine-7-sulphonic acid, or with ω-aminoethyl-β-naphthylamine-7-sulphonic acid, followed by acetylation with acetic anhydride yields a dark powder giving violet shades on wool. The acetic anhydride may be replaced by other acylating agents such as chloroacetyl chloride or toluenesulphonyl chloride, or the amino-group may be substituted in the ω-aminoethyl-2-naphthylamine-7-sulphonic acid or in the finished dye, for instance, by heating with chloroacetic acid in the presence of chalk. 2-Nitro-4-toluidine diazotised and coupled in weak mineral acid with ω-aminoethyl-2-amino-8-naphthol-6-sulphonic acid gives on acetylation a red-brown wool dye of excellent fastness to light and fulling. Similarly ω-aminoethyl-2-amino-8-naphthol-3 : 6-disulphonic acid when coupled in mineral acid solution with diazotised *p*-nitroaniline or *m*-nitroaniline or their homologues or derivatives followed by acetylation of the product gives red-brown wool dyes of good fastness to light and fulling. The dye from *p*-nitroaniline is a brown-black powder, that from *p*-aminoethylacetanilide gives a deep brown shade, whilst the corresponding dye from 5-nitro-2-anisidine in sodium bicarbonate solution, a black powder, gives beautiful olive-brown shades on wool. The corresponding dye from 4-nitro-2-anisidine gives a red-brown shade on wool. The 5-nitro-2-anisidine may be replaced by *o*-nitroaniline or its homologues and derivatives without markedly affecting the shade of the dye. The ω-aminoethyl-2-amino-8-naphthol-3:6-disulphonic acid may be acylated first and then coupled. 5-Nitro-2-aminobenzoic acid and ω-amino-

ethyl-β-naphthylamine give crystalline products dyeing acetate silk in bluish-pink shades.

R. BRIGHTMAN.

Blue monoazo dyestuffs for wool or silk. A. H. NEY, Assr. to T. HAEBLER (U.S.P. 1,586,693, 1.6.26. Appl., 13.11.24; cf. U.S.P. 874,967–8, B., 1908, 116, 117).—Diazotised *p*-aminodiphenylamine-*o*-sulphonic acid is coupled in alkaline solution with 1-acetamido-8-naphthol-3 : 6-disulphonic acid (acetyl-H-acid) to yield a dyestuff which dyes wool or silk in an acid bath brilliant reddish-blue shades which do not change in artificial light and are very fast to light, washing, and carbonising, and fast to fulling, milling, and alkali. The products from *p*-amino-*o*′-methyldiphenylamine-*o*-sulphonic acid and *p*-amino-*o*′-methoxydiphenylamine-*o*-sulphonic acid (formed by condensing *p*-chloronitrobenzene-*m*-sulphonic acid and *o*-anisidine and reducing the nitro-group) give purplish-blue and brilliant greenish-blue shades, respectively.

T. S. WHEELER.

Yellow monoazo dyestuffs. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. L. LASKA and F. WEBER (U.S.P. 1,587,005, 1.6.26. Appl., 10.9.25. Conv., 20.9.24).—Diazotised monoamines of the diphenylamine series are coupled with derivatives of 1-sulphoaryl-5-pyrazolones, such as 1-sulphoaryl-3-methyl-5-pyrazolones or 1-sulphoaryl-5-pyrazolone-3-carboxylic acids to yield dyes which dye wool yellow shades especially fast to milling. For example, *pp*′-dichloro-*o*- or -*m*-aminodiphenyl is diazotised and coupled with 1-(*o*-chloro-*p*-sulphophenyl)-3-methyl-5-pyrazolone.

T. S. WHEELER.

Bluish-green dyestuffs and colour lakes therefrom. O. STAEHLIN, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,587,435, 1.6.26. Appl., 18.12.24. Conv. 24.12.23; cf. G.P. 224,442; B., 1910, 1120).—A halogenated 1 : 5-naphthylenediaminesulphonic acid is treated with a solution of ferric chloride to give a product dyeing wool, but not cotton, greenish shades. The lakes from these dyes, *e.g.*, the barium salts of the dyes, have a more bluish tint. Owing to their greater solubility the new dyes are more readily converted into lakes than the analogous dyes from the non-halogenated 1 : 2-naphthylenediaminesulphonic acids described in G.P. 224,442 (*loc. cit.*).

T. S. WHEELER.

Manufacture of anthraquinone derivatives. BRIT. DYESTUFFS CORP., LTD., W. H. PERKIN, A. W. FYFE, and M. MENDOZA (E.P. 252,922, 8.7.25).—Better yields of the *N*-methyl-ω-sulphonic acid derivatives of 1:4- and 1:8-diaminoanthraquinone are obtained in the absence of a strong mineral acid, by the process described for diaminochrysazin (E.P. 238,717, B., 1925, 840) than by the process of E.P. 23,968 of 1899 (B., 1900, 1101), although in the case of 1 : 5-diaminoanthraquinone good results are only obtained in the presence of strong mineral acid. Little more than the theoretical amount of formaldehyde-bisulphite is required if the base is prepared in a finely divided condition by solution in sulphuric acid and dilution with water, the finely divided diamine being then washed free from acid with water. Thus 1 g.-mol. of 1 : 4-diaminoanthraquinone is dissolved in 1600 g. of sulphuric acid and after agitation for 2 hrs. at 100° is cooled and diluted with 20 litres of ice-cold water. After decanting off most of the waste acid and washing the precipitate on the filter, first with water and then with 2% soda to slight alkalinity, the paste is incorporated with a mixture of 2·5 mols. of 40% sodium bisulphite and 2·5 mols. of 40% formaldehyde. The mass is diluted with 1500 c.c. of water and stirred under a reflux for 8 hrs. at 100°, when one litre of saturated salt solution is stirred in and the ω-sulphonate filtered off after cooling. In the case of the 1:8-diaminoanthraquinone conversion is complete in about 12 hrs.

R. BRIGHTMAN.

Manufacture of anthraquinone derivatives. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 253,386, 12.8.25).—Diazotised 1-amino-anthraquinone-2-aldehyde or its halogen derivatives, either in the solid state or in solution, are treated with cuprous halides in the presence of water. The cuprous halides may be supplied as a mixture of cupric salts and reducing agents. The reaction is carried out at an elevated temperature, preferably in the absence of chlorine ions other than those of cuprous halides. Thus 10 pts. of 1-aminoanthraquinone-2-aldehyde diazotised in sulphuric acid and diluted with ice-water are run at 90—95° into the stirred solution of cuprous chloride obtained by boiling 17 pts. of copper sulphate, 8 pts. of common salt, and 7 pts. of sodium bisulphite in 1000 pts. of water until decolorised and no more sulphur dioxide is developed. The precipitated dianthraquinonyl-dialdehyde is filtered off hot and purified by extraction, *e.g.*, with acetone, or by crystallisation from high-boiling solvents. From 4-chloro-1-aminoanthraquinone-2-aldehyde treated in a similar manner 4:4′-*dichloro*-1:1′-*dianthraquinonyl*-2:2′-*dialdehyde* is obtained.

R. BRIGHTMAN.

Manufacture of benzanthrone derivatives. BRIT. ALIZARINE CO., LTD., J. ANDERSON, and W. H. DAWSON (E.P. 253,163, 10.2.25).—Benzanthrone or its derivatives such as violanthrone and *iso*violanthrone are chlorinated by solution in concentrated sulphuric acid, precipitation with water, and treatment of the fine suspension thus obtained with a soluble chlorate and hydrochloric acid in the cold. Thus 20 pts. of violanthrone are dissolved in 200 pts. of 98% sulphuric acid, and the solution is poured on to 300 pts. of ice. To this suspension 15 pts. of sodium chlorate and 103·5 pts. of concentrated hydrochloric acid (*d* 1·15) are added. After keeping for 4 hrs. the insoluble chloro-derivative is filtered off and washed. It gives bluer dyeings than the dichloro-derivative obtained by the use of sulphuryl chloride in nitrobenzene.

R. BRIGHTMAN.

Manufacture of monochloronitrobenzene. J. W. LIVINGSTON and L. P. KRYIDES (U.S.P. 1,586,253, 25.5.26. Appl., 8.8.25).—1175 pts. of

finely-divided sodium nitrate, 1600 pts. of 93% sulphuric acid, and 1240 pts. of chlorobenzene are added together in the course of 5 hrs. to an agitated mixture of 1000 pts. of 81% sulphuric acid and 200 pts. of chlorobenzene, at 110—130°. After keeping for a further hour at this temperature the mixture is poured into water. Chloronitrobenzene is obtained in satisfactory yield.

T. S. Wheeler.

Manufacture of benzanthrone derivatives. A. J. Ransford. From L. Cassella & Co. (E.P. 254,021, 27.3.25).—See U.S.P. 1,565,229 ; B., 1926, 434.

Vat [dibenzanthrone] colouring matter. Badische Anilin- & Soda-Fabr., Assees. of H. Neresheimer (U.S.P. 1,589,303, 15.6.26. Appl., 26.4.23. Conv., 16.8.22).—See E.P. 212,145 ; B., 1924, 369.

Vat dyestuff. W. Eckert and H. Greune (U.S.P. 1,588,451, 15.5.26. Appl., 17.7.25. Conv., 21.7.24).—See E.P. 237,294 ; B., 1925, 840.

Azo dyestuffs containing chromium. Soc. of Chem. Ind. in Basle, Assees. of M. Isler and L. von Mechel (U.S.P. 1,588,458, 15.6.26. Appl., 1.12.25. Conv., 28.12.23).—See E.P. 226,797 ; B., 1925, 703.

Trisazo dyestuffs. Chem. Fabr. Griesheim-Elektron, Assees. of A. L. Laska and F. Weber (U.S.P. 1,590,042, 22.6.26. Appl., 23.7.25. Conv., 15.11.24).—See G.P. 423,092 ; B., 1926, 312.

[Azo] dyestuffs containing metals. Soc. of Chem. Ind. in Basle, Assees. of F. Straub and H. Schneider (U.S.P. 1,590,482, 29.6.26. Appl., 7.4.25. Conv., 20.4.24).—See E.P. 232,620 ; B., 1925, 954.

Manufacturing perylene compounds of quinone character. A. Zinke, Assr. to F. Bensa (U.S.P. 1,590,661, 29.6.26. Appl., 7.4.25. Conv., 10.4.24).—See E.P. 232,264 ; B., 1925, 910.

Vat sulphide dyes. F. B. F. Muth and A. Schmelzer, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,590,718, 29.6.26. Appl., 16.1.25. Conv., 24.1.24).—See E.P. 243,557 ; B., 1926, 121.

Gas containing hydrogen for hydrogenation of naphthalene (F.P. 603,639).—See II.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Amidation of cotton. P. Karrer and W. Wehrli (Helv. Chim. Acta, 1926, **9**, 591—597).—Cotton fibre treated with toluenesulphonyl chloride yields a toluenesulpho-derivative which contains 1 mol. of the sulphonyl group for every 9—10 mols. of $C_6H_{10}O_5$ residue and which cannot be dyed with acid dyes, and this on treatment with aqueous ammonia yields, slowly at ordinary temperature and more rapidly at 100°, an amidated fibre which contains 1 mol. NH_2 for every 9—11 mols. of $C_6H_{10}O_5$ residue, ammonium toluenesulphonate separating during the reaction. The amidated fibre is readily dyed with acid dyes, and shows satisfactory results with regard to fastness, the best results being obtained with dyes of the after-chrome, pyrazolone, and anthraquinone acid groups, and less satisfactory with those of the azo and triphenylmethane groups. Similar results are obtained using methyl-, dimethyl-, and ethylamines, and to a lesser extent, with benzylamine, aniline, and α-naphthylamine, but the tertiary bases trimethylamine and pyridine give better results than does ammonia. Amidated products can also be obtained from the fibre previously treated with benzenesulphonyl and disulphonyl, naphthalenesulphonyl, and ethylsulphonyl chlorides, but not from cellulose acetate, benzoylated cotton, or nitrocellulose. The action of the acid dyes is due to salt formation with the amino-group, but in fibres of albuminous origin the nitrogen is present as acid amides and has no basic properties, and it is shown that fibre treated with acetyl- or benzoyl-sulphanilic acid chloride is not dyed with acid dyes, but if it is treated with p-nitrobenzoyl chloride followed by the reduction of the nitro-group to the amino-group with ammonium sulphide, it is then readily dyed in this manner.

J. W. Baker.

Action of solutions of sodium sulphide on linen fibres. P. P. Victorov (Textilber., 1926, **7**, 61—63, 444—445).—A comparison of the purifying action of boiling solutions of sodium sulphide and caustic soda on raw linen yarns containing 83·8% of pure cellulose. Raw linen yarns digested with boiling water and a solution containing 10% (calc. on the weight of yarn) of sodium sulphide suffered losses of 3·8% and 6·4% respectively. Solutions of caustic soda effect the removal of impurities (pectic and other incrusting substances) from raw linen more rapidly than solutions of sodium sulphide of equal concentration (calc. on the weight of linen), but solutions of caustic soda produce greater deterioration of linen, especially when used for prolonged periods at high temperatures. The lustre, handle, and whiteness of bleached linen yarns previously scoured with sodium sulphide are not inferior to those of similar yarns previously scoured with caustic soda. A. J. Hall.

Action of acids on artificial silks. K. Wolfgang (Kunstseide, 1926, **8**, 175—177).—The brittleness occasionally found in artificial silks is due to the effects of acids remaining in the silk from processes of manufacture. Samples of viscose, cuprammonium and nitrocellulose silk yarn of 120 denier impregnated so that they contained their own weight of various solutions of sulphuric, formic, acetic, and lactic acids, then dried for 1 hr. at 90°, and afterwards conditioned in air for 24 hrs. suffered losses of tensile strength exceeding 5% when the concentrations of the acid solutions were greater than 0·05%, 0·3%, 0·3%, and 1·5% respectively.

A. J. Hall.

Increasing the wet strength of [cellulose] artificial silks. W. Bruckhaus (Kunstseide, 1926, **8**, 115—116).—Artificial silk yarn or fabric is impregnated with a solution containing 2 kg. of alum,

2·5 kg. of 3% lactic acid, 10—12 kg. of 4% formaldehyde, and 10—35 litres of water, then hydro-extracted, dried at 60°, soaped in a solution containing 5—7 g. of Marseilles soap per litre, washed, scoured in a 0·3% solution of acetic acid, and dried at a low temperature. The treated silk has an increased wet strength, is more resistant to alkalis, and has a greater affinity for dyes. A. J. HALL.

Effect of heat upon cellulose. J. W. BAIN and G. M. CHUTE (Trans. Roy. Soc. Canada, 1926, [iii.], **20**, III., 189—191 ; cf. B., 1925, 349).—The presence of dextrose in cotton which has been heated at 200° is definitely established. Other hexoses, pentoses, and methylpentoses are probably absent. Some formic acid and small amounts of acetic acid are formed during the heating. J. S. CARTER.

Ripening of viscose. R. O. HERZOG (Papier-Fabr., 1926, **24**, Fest- u. Ausland-Heft, 94—97 ; cf. B., 1925, 6).—The chemical changes which characterise the process of "ripening" of viscose are accompanied by physical changes in the colloidal condition, which undergoes a slow transition from the sol to the gel state. These may be defined as a gradual coagulation through the formation of secondary particles from the original primary particles, a process which should conform to a bimolecular reaction. The degree of ripeness is determined empirically by Hottenroth's method by titration with ammonium chloride, and the quantity of ammonium chloride may be expressed in terms of a function of the time of ripening by the formula $at=b-q$, a and b being constants. The coagulation by ammonium chloride is determined by chemical changes in the xanthate, dehydration of the colloid particles, increase in size of the colloid particles, *i.e.*, relative lowering of concentration. In addition reaction equilibria between the electrolytes already present and those added, and adsorption reactions also exert an influence. A further physical change which accompanies the ripening is the decrease in extensibility or increase in the modulus of elasticity of the regenerated thread. Thus the extensibility may also be expressed empirically as a function of the ammonium chloride titration value, the determined results for the modulus of elasticity agreeing well with those calculated by the formula $1/\sqrt{D}=H-J\rho$, H and J being constants. Lastly, the ripening process is accompanied by an increase in viscosity of a peculiar nature (cf. *loc. cit.*) which involves the gradual formation of a structure in the solution, becoming increasingly more stable towards vigorous agitation. The optimum period for spinning occurs at a ripeness degree of 7, when this incipient gelation or structure is very easily destroyed but rapidly re-forms. This alteration is due to a change in the size or shape of the particles, presumably only the secondary particles, which probably assume a rodlet formation (series of micellæ). J. F. BRIGGS.

Chemistry of wood. VIII. Further studies of sapwood and heartwood. IX. Spring wood and summer wood. G. J. RITTER and L. C. FLECK (Ind. Eng. Chem., 1926, **18**, 576—577, 608—609 ; cf. B., 1923, 9 A, 1123 A).—Further analyses of sapwood, heartwood, spring wood, and summer wood are given for various American woods. In soft woods the water, ether, and alkali extracts are higher and the cellulose and lignin correspondingly lower in the heartwood than in the sapwood ; white spruce, however, shows practically the same cellulose and lignin content in both bands of growth. The hardwoods may be divided into two groups, those with high extractives (water, ether, and alkali extracts) and low cellulose content in the sapwood, and those with high extractives and low cellulose in the heartwood. Spring wood shows a consistently higher content of lignin together with a lower content of cellulose (except in the case of loblolly pine and red alder) than does summer wood. No other outstanding differences were noted, but there is a tendency for spring wood to contain more extractives and more pentosans both in the wood and in the cellulose isolated therefrom. D. J. NORMAN.

Fluorescence of sulphite-cellulose. C. W. LEUPOLD (Papier-Fabr., 1926, **24**, 397—398).—The fluorescence of sulphite-cellulose waste lye is due to colloidal sulphur, and it is therefore possible that the fluorescence of sulphite-cellulose is due to sulphur compounds. This view is supported by the fact that the fluorescence is destroyed by treatment with chlorine or iodine, which would decompose any thio-acids or polythionic acids. A. GEAKE.

Freeness of sulphite pulp. D. S. DAVIS (Ind. Eng. Chem., 1926, **18**, 631—633).—The operation of the Williams freeness tester (Pulp and Paper Mag., 1925, **23**, 443) is described. Graphs and equations are given for converting freeness numbers for one orifice into freeness numbers for another of a different size ; also for converting the freeness value at any known concentration between 0·3 and 0·5% into freeness at standard concentration (0·4% of bone-dry pulp). The observed freeness of mixed stock does not agree with the freeness value calculated from the proportions and freeness of the components of the mixture ; a graphic method is outlined for computing the one value from the other. The mathematical treatment of the subject is of general application. D. J. NORMAN.

See also A., July, 673, **Adsorption of alkali hydroxide by cellulose** (LIEPATOV). 677, **Viscosity anomalies in cellulose sols** (VON NEUENSTEIN) ; **Structure of cellulose and its significance in chemical transformations** (HERZOG). 715, **Cryoscopic behaviour of crystalline cellulose acetates** (HESS and SCHULTZE).

Fermentation products from cellulose. SPEAKMAN.—See XVIII.

PATENTS.

Operating spinning machines for artificial silk. WERDOHLER PUMPENFABR. P. HILLEBRAND (E.P. 248,715, 26.1.26. Conv., 4.3.25).—In the ordinary type of artificial silk spinning machine, in which the liquid is fed to the spinnerets through a pressure-equalising chamber containing compressed

air, the liquid both enters and leaves the chamber at the bottom, with the result that the surface stratum of liquid in contact with the air remains unchanged and gradually hardens, thereby impairing the pressure-balancing functions of the device. This is obviated by introducing the spinning solution into the chamber at a point above the level of the liquid therein. D. J. NORMAN.

Nozzles for production of artificial filaments. C. L. WALKER (E.P. 253,209, 14.3 and 15.10.25).—Very fine tubes of platinum or platinum–iridium are sealed with glaze into holes in a porcelain nozzle, or are fused into a glass nozzle. Platinum rivets may be similarly sealed and then drilled with a fine hole. The tubes are formed from a rod through which a hole is bored and filled with a base metal ; the rod may then be drawn out to the desired diameter and the base metal dissolved out. Tubes with bores of any desired shape may be similarly produced. A. GEAKE.

Manufacturing artificial silk and other products from nitrocellulose. E. BINDSCHEDLER and G. JUER, Assrs. to TUBIZE ARTIFICIAL SILK CO. OF AMERICA (U.S.P. 1,584,005, 11.5.26. Appl., 1.7.24).—Alcohol–ether solutions of nitrocellulose are spun into a coagulating bath comprising one or more metal halides dissolved in dilute alcohol. D. J. NORMAN.

Plastic compounds of acetylcellulose. PATHÉ CINEMA, ANC. ETABL. PATHÉ FRÈRES (E.P. 237,900, 22.7.25. Conv., 4.8.24).—Plastic compounds are prepared by incorporating with cellulose acetate a mixture of triphenyl phosphate and tricresyl phosphate with the assistance of a solvent. Suitable proportions are : 100 pts. of cellulose acetate, 15 of triphenyl phosphate, 12 of tricresyl phosphate. The use of such a mixture avoids the extrusion of crystals on the one hand and oily globules on the other. J. F. BRIGGS.

Reducing the viscosity of nitrocellulose. L. E. BRANCHEN, Assr. to EASTMAN KODAK CO. (U.S.P. 1,588,089, 8.6.26. Appl., 1.9.25).—Nitrocellulose is softened with a penetrant liquid and then treated with acid. A. GEAKE.

Extraction of cellulose. E. R. CLARK, Assr. to M. S. ERLANGER (U.S.P. 1,587,631, 8.6.26. Appl., 12.2.25).—Chlorine is supplied to water which is circulated by an external pump through cellulose-containing material. A. GEAKE.

Making pulp from [rice] hulls. J. F. and H. F. J. PUTTAERT (U.S.P. 1,588,335, 8.6.26. Appl., 12.2.20).—Rice hulls are boiled with dilute sodium hydroxide solution for 6—8 hrs. at a pressure of 20—80 lb. per sq. in., removed, washed, and " defibercated." A. GEAKE.

Bleaching of paper pulp. R. B. WOLF (E.P. 251,205, 17.12.25. Conv., 28.7.25).—Air is directed on to the surface of the pulp during the bleaching operation, firstly to control the temperature of the mass, thereby facilitating the bleaching of relatively thick stock, *i.e.*, containing more than 10% of fibre, and secondly to remove volatile decomposition products and thus accelerate the bleaching operation. A suitable apparatus consists of a vertical cylindrical tank provided with a central tube fitted internally with a screw conveyor for carrying the pulp to the top of the tube, where it is distributed over an apron and drops back into the tank. A scraper is provided at the bottom of the tank to ensure complete circulation of the pulp through the central tube. Jets of air play on the pulp as it passes in a thin layer over the apron ; the amount of air introduced depends on its temperature and humidity and also on the degree of cooling it is desired to effect. For instance, in a tank with a capacity of 4 tons, with the materials entering at 30°, air should be introduced at 4000 ft. per min. through about 30 nozzles of approximately 3 in. diameter at their outer ends. D. J. NORMAN.

Sizing cellulose fibres [paper pulp]. J. A. DE CEW (U.S.P. 1,585,469, 18.5.26. Appl., 15.12.25).—The fibres are treated in the beater with aluminium sulphate, and a stream of size emulsion is added to the pulp as it is entering the Jordan engine (refiner). J. F. BRIGGS.

Manufacture of sand paper or like composite structures. MINNESOTA MINING AND MANUF. CO., Assees. of F. G. OKIE (E.P. 226,803, 9.12.24. Conv., 29.12.23 ; cf. E.P. 194,274 and 219,341 ; B., 1924, 905).—A suitable binder for flexible, waterproof sand-paper consists of turpentine, rosin, or other similar resin, and tung oil or other similar oil, *e.g.*, tung oil 53%, hardened rosin 12%, linseed oil 10%, drier 1%, and thinner 24%. The size to fix the abrasive is of similar composition to the binder, but with more thinner, and hence adheres strongly to it. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 211,203 and 209,872.) A. GEAKE.

Safety paper. A. I. CONE (U.S.P. 1,584,850, 18.5.26. Appl., 6.5.25).—The paper contains a mercurous salt. J. F. BRIGGS.

Apparatus for sizing, dyeing, and impregnating paper. B. KREMLER (G.P. 427,531, 8.11.23).—The paper passes between rollers the channels of which may be connected or disconnected with a circulating apparatus for the liquid. Uniform sizing is obtained with a minimal volume of liquid. A. GEAKE.

Making a new type of cellulose derivatives. L. LILIENFELD (U.S.P. 1,589,606, 22.6.26. Appl., 20.3.22. Conv., 2.4.21).—See E.P. 177,810 ; B., 1924, 11.

Making cellulose derivatives [ethers]. L. LILIENFELD (U.S.P. 1,589,607, 22.6.26. Appl., 20.3.22. Conv., 2.4.21).—See E.P. 177,809 ; B., 1924, 11.

Manufacture of coloured cellulose plastics and solutions thereof. BADISCHE ANILIN- & SODA-FABR., Assees. of C. IMMERHEISER, C. NEUBAUER,

and E. SCHARF (U.S.P. 1,589,700, 22.6.26. Appl., 29.11.24. Conv., 26.6.24).—See E.P. 247,288; B., 1926, 315.

Bleaching vegetable fibres (E.P. 251,014).—See VI.

Recovering calcium carbonate from caustic liquors (U.S.P. 1,587,679).—See VII.

Gelatinising nitrocellulose (E.P. 252,978).—See XXII.

Bactericide and fungicide from sulphite-cellulose waste lye (U.S.P. 1,585,792).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

[Influence of after-treatments on the] fastness to light of dyeings obtained from Naphthol AS compounds. L. LÖSCHER (Textilber., 1926, 7, 243—244).—Samples of bleached cotton fabric dyed in the usual manner with Naphthol AS+Fast Red GL base, Naphthol AS—SW+Fast Red KB base, and Naphthol AS—RL+Fast Red RL base, and then subjected to various treatments with cold and hot water, and warm and boiling solutions of soap, were exposed for about 2½ summer months to sunlight, one series of exposures being protected by glass. The dyeings not protected by glass faded somewhat more rapidly, but both series faded similarly. Generally, fastness to light of the dyeings described above is proportional to the severity of the soaping treatment and is independent of free naphthols or bases which may be present. Severe soaping is necessary for obtaining maximum fastness of the combinations Naphthol AS+Fast Red GL base and Naphthol AS—SW+Fast Red KB base, but Naphthol AS—RL+Fast Red RL base requires but a moderate treatment with a warm soap solution. Maximum fastness to light of the combination Naphthol AS+Fast Red GL base is secured by soaping at boiling temperature for 5 min. Similarly Naphthol AS—SW+Fast Red KB base and Naphthol AS+Fast Red RL base requires treatment for ½ hr. at boiling temperature, the shade of the last-named combination being thereby rendered bluer. After washing with cold water only, Naphthol AS—RL+Fast Red RL base has the same fastness as Naphthol AS+Fast Red GL base after soaping for ½ hr. at boiling temperature. A. J. HALL.

Printing cellulose acetate silk. A. SCHNEEVOIGT (Textilber., 1926, 7, 354).—Cellulose acetate silk may be printed satisfactorily with alkaline pastes containing vat dyes (saponification of the silk thereby occurs without loss of lustre) or by the usual methods after previous superficial saponification of the silk. After immersion in water and squeezing under comparable conditions, fully, partially (as desirable for printing purposes), and non-saponified cellulose acetate silk fabrics retain 74%, 50%, and 35% of water respectively. Basic dyes are equally fast on superficially saponified cellulose acetate and cotton. Azonine, Azanile, Silkone, and Azole dyes are not satisfactory for printing purposes since in their subsequent development by diazotisation and coupling, white effects are very easily stained. Very satisfactory effects on cellulose acetate silk are obtained by printing with a paste containing 50 g. of a suitable anthraquinone dye (*e.g.*, Yellow 3G extra, Yellow R, Rosa R, or Violet B), 50 g. of Turkey-red oil, 50 g. of glycerol, 50 g. of Ludigol (*m*-nitrobenzenesulphonic acid), and 800 g. of a thickening containing starch and British gum, and subsequently steaming for 5 min. in a Mather–Platt (loss of lustre occurs when steaming is effected under pressure). The addition of resorcinol to discharge pastes containing Rongalite allows the production of satisfactory white discharges of basic dyes. A. J. HALL.

[Use of] Aktivin in [textile] printing. R. WEGENER (Textilber., 1926, 7, 446).—The stability of Aktivin (sodium *p*-toluenesulphochloramide) renders it more suitable than other well-known oxidising or bleaching agents for treating fabrics before or after printing. For example the chlorination of woollen fabric before printing is satisfactorily replaced by treatment for 20—30 min. in a cold solution containing per litre, 1 g. of Aktivin and 5 c.c. of hydrochloric acid of 21° B. (*d* 1·17), the fabric being subsequently treated with an "antichlor," *e.g.*, a 1% solution of sodium thiosulphate. For clearing the whites of printed goods treatment with a solution containing per litre, 0·2 g. of Aktivin and 0·2 g. of formic acid (for fabrics printed with basic dyes and other dyes sensitive to chlorine) or 1 g. of Aktivin and 0·5 g. of formic acid or 0·6 g. of hydrochloric acid of *d* 1·17 (for fabrics printed with fast vat dyes) is satisfactory. Aktivin is suitable for the preparation of starch pastes for thickening printing pastes. A satisfactory paste is obtained by heating 2 kg. of potato starch, 15 g. of Aktivin, and 20 litres of water for 10 to 15 min. and adding to the product half of its weight of a 7% solution of gum tragacanth. Coloured effects obtained by printing with vat dyes may frequently be improved by after-treating the fabric for 10 min. with a warm solution containing per 200 litres, 100 g. of Aktivin and 100 g. of 80% formic acid. The use of standing baths containing Aktivin is possible. A. J. HALL.

Red and white discharges on dark indigo bottom. J. POKORNY (J. Soc. Dyers and Col., 1926, 42, 157—158; cf. B., 1926, 437).—Red discharges are obtained without tendering the fabric by printing cotton fabric prepared with β-naphthol or β-naphthol R (a mixture of 9 pts. of β-naphthol and 1 pt. of F-acid) with a paste containing 20 pts. of lead chromate, 40 pts. of starch–tragacanth thickening, 28 pts. of diazotised *p*-nitroaniline, and 12 pts. of water, afterwards passing the material during 3—5 sec. at 37° through hydrochloric acid of *d* 1·135 containing 30 g. of ferrous sulphate per litre, then steaming it at 35° for 35—40 sec., washing it with water, and soaping. The hydrochloric acid should not be stored in a lead-lined trough since the presence of lead acts unfavourably in the process of discharge. The printing paste requires a rather deep engraving, 22—24 lines being used instead of the usual 27—28 on the printing roller. A. J. HALL.

Amidation of cotton. Karrer and Wehrli.—See V.

Liquefying action of various substances on starch. Haller and Hohmann.—See XVII.

Patents.

[Compositions for] scouring of textile fibres. L. L. Lloyd, A. Womersley, C. Wilkinson, and A. Scott (E.P. 252,811, 10.3 and 10.12.25).—Hydronaphthalene (*e.g.*, tetra- or hexa-hydronaphthalene)-sulphonic acids, or condensation products of hydronaphthalenes or hydronaphthalenesulphonic acids and fatty acids, such as oleic acid or the acids derived from castor, arachis, olive, or cottonseed oils, are satisfactory substitutes for soaps in the scouring of wool, and they may be used at higher temperatures (*e.g.*, at 60° instead of 50° for botany wool) than is possible with soap and alkalis without producing deterioration of the wool. A. J. Hall.

Kier solution controller. A. F. Taylor (U.S.P. 1,584,491, 11.5.26. Appl., 7.7.25).—The vertical kier is fitted centrally at the bottom with a block; this block is provided with an axial passage and radial passages communicating therewith, and acts as a support for a perforated false bottom which divides the kier into an upper and a lower compartment. A perforated tube closed at the top and communicating at its lower end with the upper end of the axial passage in the block extends into the upper compartment. D. J. Norman.

Rapid bleaching process for loose or worked-up vegetable fibres of any kind. Chem. Fabr. Griesheim-Elektron, C. G. Schwalbe, and H. Wenzl (E.P. 251,014. 22.1.25).—The material, optionally after a preliminary treatment with hot, very dilute acid, *e.g.*, 0·1% acetic acid, is drenched with concentrated bleaching liquor containing at least 20 g. (20—30 g.) of available chlorine per litre, and squeezed until it retains sufficient liquor to effect the desired degree of bleaching, the temperature being maintained above 30°, *e.g.*, 30—40°, by pre-heating either the material or the bleaching solution. Loose fibrous material such as paper pulp is preferably bleached in a centrifuge and washed while the centrifuge is running: where other methods are adopted the spent bleach liquor must be displaced by water as far as possible without dilution. In this way colloidal and other impurities capable of causing yellowing are not precipitated on the bleached pulp as is the case when the spent liquor is diluted before removal. When the material to be bleached is in the form of a continuous web, it may be supported on a conveyor, the speed and length of travel being so arranged that the material, after saturation with bleaching solution, is exposed for about 5 min. to a moist warm atmosphere. The use of concentrated solutions of bleaching agents under these conditions has, it is claimed, no deleterious effect on the fibre. D. J. Norman.

Dyeing hanks. P. Missy (U.S.P. 1,586,377, 25.5.26. Appl., 2.9.25).—Dye solutions are sprayed by horizontally opposed jets on to hanks of yarn suspended on rotating rollers. L. A. Coles.

Production of pattern and similar effects on fabrics containing cellulose. R. Clay, Ltd., H. M. Scott, and L. Thompson (E.P. 251,102, 1.5.25).—A permanent linen-like effect, not characterised by transparency, is imparted to cellulose fabrics, particularly cotton, by calendering the resist-printed fabric prior to treating it with a parchmentising agent. This process may be applied to mercerised or unmercerised fabric and any suitable reagent, *e.g.*, sulphuric acid, zinc chloride, nitric acid (*d* 1·46), etc., having a parchmentising action may be used. For example, heavy bleached cotton fabric is printed with a resist consisting of a 10% solution of gelatin, dried, and conditioned to contain 5% of moisture. It is then calendered at a pressure of 0·75 ton per linear in., the metal calender bowl being heated to 190°. The cooled fabric is subsequently exposed for 5 sec. at 13° to sulphuric acid (*d* 1·59), washed, and finished in any desired manner. D. J. Norman.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Titration of arsenious acid with permanganate. O. Cantoni (Annali Chim. Appl., 1926, **16**, 153—156).—The reaction between arsenious acid and permanganate, which proceeds slowly under ordinary conditions, becomes rapid and complete, even in the cold, in presence of a small amount of potassium iodide. Hence, if allowance is made for the slight quantity of permanganate used in liberating iodine from the iodide, titration with permanganate furnishes a convenient method of determining arsenious acid. To a little sulphuric or, in absence of iron compounds, hydrochloric acid are added a drop of dilute potassium iodide solution and then very dilute permanganate solution until the liquid turns pink. The arsenious acid solution to be examined is then added, and the liquid titrated with permanganate until the pink coloration reappears. T. H. Pope.

Large crystals in sulphate of ammonia manufacture. H. J. Hailstone (Gas J., 1926, **174**, 646—648).—The production of large crystals of ammonium sulphate depends on the maintenance of correct physical conditions within the saturator for a sufficient time to allow the crystals to grow (a question of design), and the maintenance of unvarying low acidity (a matter of control). Free circulation of the liquor is desirable and a saturator of ample size free from corners and large walls is best, so that the composition of the liquor is uniform. Further refinements in design are suggested. Control is usually carried out by the hydrometer. This is affected by the difficulty of obtaining a true sample and cannot be made automatic under the conditions of practice. A better method might be worked out by means of a temperature record (a fall in acidity being accompanied by a slight fall in temperature), or by the continuous determination of acidity by measurement

of the refractive index or electrical conductivity of the liquor. The rapid change in the conductivity of sulphuric acid solutions between 0 and 5% concentrations makes the latter especially hopeful. For control of the acid feed a lead feed regulator such as is used on Gay-Lussac towers is recommended. It consists of a box with an oblique line of small holes in the side, the number of holes through which acid is flowing being a measure of the feed. C. IRWIN.

Ammonium sulphate and sodium sulphate from sodium bisulphate and ammonia. H. MOLITOR (Chem.-Ztg., 1926, 50, 485—486).—Commercial sodium bisulphate (nitre-cake) is dissolved in water to give a solution (*d* 1·325) which is treated with ammonia or ammoniacal gases in closed lead-lined iron vessels arranged in series. When the iron present in the solution in each vessel is completely precipitated the solution is removed and filtered, and the filtrate is evaporated in steam-jacketed pans or in an evaporator until the volume is reduced to one-half. In this way 95% of the sodium sulphate is precipitated as the anhydrous salt free from iron and ammonium compounds. The precipitate is separated hot from the mother liquor on a vacuum filter, and washed with steam in a centrifuge to obtain pure anhydrous sodium sulphate in powder form. The mother liquor is further evaporated until crystals begin to form, then allowed to cool. The ammonium sulphate precipitate is treated as usual; it contains some sodium sulphate, but this does not affect its use as a fertiliser. The iron hydroxide obtained in the process yields a red pigment when heated to 700° with sodium hydroxide. A. R. POWELL.

Comparison of methods for the determination of sodium sulphide. P. P. BUDNIKOV (J. Russ. Phys. Chem. Soc., 1925, 57, 207—214).—The most satisfactory methods for the determination of sodium sulphide are: (1) the iodometric method in which excess of iodine is added and the excess titrated with thiosulphate (B., 1923, 1067A), and (2) Podreschetnikoff's method (B., 1908, 21). T. H. POPE.

Oxidising properties of sulphur dioxide. W. WARDLAW (J.S.C.I., 1926, 45, 210—214 T).—Sulphur dioxide oxidises a solution of ferrous chloride in concentrated hydrochloric acid in accordance with the equation:—

$$4FeCl_2+SO_2+4HCl=4FeCl_3+2H_2O+S.$$

This explains why ferric chloride is incompletely reduced by sulphur dioxide in the presence of concentrated acid. Similarly, cuprous chloride is oxidised by sulphur dioxide in the presence of hydrochloric acid of concentration as low as 1·5*N*. The quantitative reduction of cupric to cuprous chloride takes place only under very special conditions. The fact that the oxidation of sulphur dioxide by mercuric chloride, which takes place as follows:—

$$2HgCl_2+SO_2+2H_2O=Hg_2Cl_2+2HCl+H_2SO_4$$

is only quantitative when the solution contains not more than about 2 g. of sulphur dioxide per litre, is another instance of the influence of acid concentration on the properties of sulphur dioxide. In the presence of phosphoric acid, sulphur dioxide is also an oxidising agent, for it readily oxidises ferrous phosphate to the ferric condition.

Physico-chemical and electrochemical aspects of sulphur dioxide as an oxidising agent. S. R. CARTER (J.S.C.I., 1926, 45, 207—210 T).—Determinations of oxidation-reduction potentials show that an increase in acid concentration raises the oxidising power of sulphur dioxide. On the other hand, ferrous-ferric salts become better reducing agents in hydrochloric and phosphoric acids, respectively. The potentials of the cuprous-cupric salts are less than those of the ferrous-ferric salts in solutions of low acid concentration and they give a maximum in 6*N*-hydrochloric acid. Observations on reaction velocities as well as electrolytic experiments suggest that the reduction of sulphur dioxide does not proceed to sulphur directly. It is probable that an intermediate compound X is formed reversibly and that this reaction is followed by a second in which X decomposes irreversibly giving sulphur.

See also A., July, 670, **Vapour pressure of hydrogen cyanide** (SINOZAKI, HARA, and MITSUKURI); **Vapour tension of carbonyl chloride** (GERMANN and TAYLOR). 675, **Viscosity of aqueous solutions of sodium silicates** (MAIN). 684, **Thermal decomposition of siderite, rhodochrosite, and dolomite** (HEDVALL). 685, **Thermionic properties of mixtures used as catalysts in synthesis of ammonia** (KUNSMAN); **Heat of formation of calcium cyanamide** (EHRLICH); **Pure calcium carbide and its heat of formation** (RUFF and JOSEPHY). 691, **Reduction of ferric salts with hydrogen sulphide** (MOLDENHAUER and MISCHKE). 692, **Reactions in the solid state** (BALAREV); **Thermal decomposition of carbonates** (BRUZS). 695, **Reactions of solid alkaline-earth oxides with sulphides, carbides, silicides, and phosphides** (HEDVALL and NORSTRÖM). 696, **Oxidation of ammonia** (PARTINGTON). 697, **Oxides of chromium** (SIMON and SCHMIDT). 699, **Preparation of oxygen-free nitrogen** (KAUTSKY and THIELE). 701, **Potentiometric titration of bromide and chloride** (LANGE and SCHWARTZ). 702, **Volumetric determination of hypophosphorous acid and hypophosphites** (KÖSZEGI). 704, **Precipitation of aluminium as hydroxide by means of ammonia** (JANDER and RUPERTI).

Recovery of sulphur from waste gases from metallurgical furnaces. WILL.—See X.

PATENTS.

Manufacture of phosphoric acid and generator gas. (A) W. KYBER, Assee. of E. BRITZKE; (B) W. KYBER (E.P. [A] 242,650, 4.11.25, and [B] 248,322, 14.12.25. Conv., 7.11.24 and 25.2.25).—(A) Phosphorites are heated with silicates and carbon in a shaft furnace, the evolved gases being treated in a recuperator or Cowper stove at a temperature of 1000—1300° by addition of air, whereby the carbon dioxide formed by oxidation acts upon the phosphorus vapour in accordance

with the equation, $2P+5CO_2=P_2O_5+5CO$. The phosphorus pentoxide is separated from the mixture of gases, and the residual gases consisting chiefly of carbon monoxide can be used as a generator gas. (B) Gases containing carbon dioxide, *e.g.*, flue-gases or lime-kiln gases, may be substituted for air in the process described under (A). H. ROYAL-DAWSON.

Producing concentrated phosphoric acid. F. G. LILJENROTH (E.P. 252,953, 22.9.25).—In the method of producing phosphoric acid by oxidation of phosphorus by steam at a high temperature, with or without a catalyst, a large excess of steam is necessary to complete the reaction, and with a simple condensation of the reaction products the acid produced is dilute. Concentrated acid can, however, be obtained by fractional condensation of the vapours, for instance, by water sprays in towers packed with Raschig rings. The first tower works at 100° and condenses strong acid; the second at about 44° condenses any unoxidised phosphorus as liquid, while the last is kept as cool as possible to dry the hydrogen which is the other reaction product (cf. G.P. 406,411 and 409,344; B., 1925, 242, 448). C. IRWIN.

Recovery of hydrochloric acid from ferrous chloride liquor. F. KRAUS (G.P. 427,538, 8.7.25).—The hot liquor is poured on to hot ferric oxide, and the mixture is passed through one or more muffle furnaces, a portion of the residual ferric oxide being used again and the remainder withdrawn from the furnace. L. A. COLES.

Manufacture of hydrochloric acid from chlorine and steam. B. NEUMANN, W. STEUER, and R. DOMKE (G.P. 427,539, 4.4.25).—A mixture of chlorine and steam is passed over heated coke or carbonaceous material containing iron oxide or other iron compounds as catalysts, produced, *e.g.*, by carbonising material containing iron. L. A. COLES.

Purifying pearl essence. J. PAISSEAU (E.P. 240,858, 2.10.25. Conv., 3.10.24).—Pearl essence with its impurities in the form of an aqueous paste is agitated with a liquid immiscible with water, but capable of wetting the particles of pearl essence. Amyl acetate, petroleum spirit, aromatic hydrocarbons, and similar liquids are all suitable, but the risk of an emulsion forming renders it desirable to add a varnish or viscous substance to the liquid, *e.g.*, cellulose acetate in tetrachloroethane may be used. The purified pearl essence is decanted with the organic liquid and separated by settling or centrifuging. The impurities, which are preferentially wetted by water, remain in the aqueous layer. C. IRWIN.

Manufacture of xanthates. BRIT. DYESTUFFS CORP., C. J. T. CRONSHAW, and W. J. S. NAUNTON (E.P. 252,500, 27.3.25).—A solution of an alkali hydroxide is added slowly to a well agitated mixture containing theoretical amounts of alcohol (as methylated spirit, or, in the case of higher homologues, the constant-boiling mixture of the alcohol and water), and carbon disulphide in a reaction vessel provided with a reflux condenser. After a few hours any unchanged alcohol and carbon disulphide are recovered by distillation, and the crude alkali xanthate is dissolved in water and treated with sufficient dilute hydrochloric acid to neutralise any free alkali and to decompose any alkali thiocarbonate that may be present. To this solution is added, while stirring, the theoretical amount of a solution of a soluble salt of a metal which gives an insoluble xanthate, the latter being precipitated in a state of division such that, after washing, it is obtained in a high state of purity. H. ROYAL-DAWSON.

Treating crude cyanide. R. E. GLOVER, JUN., and T. H. ROGERS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,586,509, 25.5.26. Appl., 12.3.21).—Solutions of crude cyanide prepared from calcium cyanamide are not suitable for some processes and are liable to decomposition because of the calcium present. Precipitation of this calcium with sodium carbonate in the ordinary way is not satisfactory as loss of cyanide always occurs. If the crude cyanide is mixed with sodium carbonate and this mixture added to water, a little at a time, the calcium is only momentarily in solution and is thrown down in a form which can be easily removed. E. S. KREIS.

Producing nitrogen compounds. E. S. MATTHEWS (U.S.P. 1,586,823, 1.6.26. Appl., 29.8.17. Renewed 23.10.25).—Air is heated under pressure and the heat abstracted from it while it is under pressure is used for compressing more air and for circulating the fluids. Water is used to cool the mixture and the water so warmed is led through the reaction products to remove the nitrogen oxides formed. E. S. KREIS.

Recovering calcium carbonate from caustic liquors. G. A. RICHTER and S. L. SWASEY, Assrs. to BROWN Co. (U.S.P. 1,587,079, 8.6.26. Appl., 17.1.24).—A solution of the sodium compounds recovered from black liquor from soda-cellulose manufacture is treated with insufficient lime to causticise it completely. After removing the precipitate the solution is again causticised with lime and the precipitate recovered. A. GEAKE.

Decomposition of insoluble thio salts [thioantimonates]. N. E. WILSON (Can. P. 252,563, 11.6.24).—Solid thioantimonates are treated with mineral acids capable of decomposing them, antimony pentasulphide being precipitated. L. A. COLES.

Manufacture of aluminium sulphate. DORR Co., Assees. of H. N. SPICER (Can. P. 253,301, 5.3.25).—Solid material containing aluminium is digested with sulphuric acid, and the residue is separated from the solution by sedimentation at a uniform temperature and under such conditions that the liquid is retained in contact with the solid only for a short time. L. A. COLES.

Production of carbon dioxide and nitrogen compounds [ammonia]. CANADIAN PRESS-AIR LTD., Assees. of H. W. JOHNSTON and E. J. POPE (Can. P. 254,386, 15.4.25).—Nitrogen obtained by passing air over red-hot coke and subsequent removal of carbon dioxide, is combined with electrolytic hydrogen. L. A. COLES.

Production of magnesium hydroxide. R. MONTERUMICI (F.P. 600,396, 7.7.25. Conv., 19.7.24).—Solutions containing magnesium salts are stirred with ammonia at the lowest practicable temperature, or, alternatively, the solid salts are treated with ammonia. L. A. COLES.

Conversion of soluble sulphides into chlorides and sulphur. A. JAHL (F.P. 603,141, 14.9.25. Conv., 3.7.25).—The sulphides are treated with hydrochloric acid and an excess of sulphur dioxide. L. A. COLES.

Production of salt from brine. A. WITTIG (G.P. 427,782, 23.1.23).—Brine is cooled and a portion of the water is simultaneously evaporated by subjecting it to reduced pressure, and heat generated by compressing the liberated vapour is used for again heating the liquid. L. A. COLES.

Decomposition of aluminium silicates. K. HEPKE (G.P. 427,806, 26.3.24).—Aluminium silicates are heated to 400—600° with kieserite and rock salt in such quantities that the product contains alumina in addition to sodium sulphate and magnesium silicate. L. A. COLES.

Treatment of barium peroxide for obtaining hydrogen peroxide and an improved *blanc fixe*. I. E. WEBER, B. LAPORTE, LTD., and H. E. ALCOCK (E.P. 252,768, 2.3.25).—Wet hydrated barium peroxide is decomposed with phosphoric acid (*d* 1·12), and the mixture is passed through a filter-press to separate the barium phosphate, the hydrogen peroxide solution being collected. After washing the barium phosphate, it is dissolved by agitation with phosphoric acid (*d* 1·12) ; the impurities mostly remain undissolved. The solution is filtered, heated to 60—80°, and treated with sulphuric acid to precipitate *blanc fixe*, which is separated and mixed with barium carbonate to neutralise any free acid, before being washed and dried. The phosphoric acid is used again in the process. H. ROYAL-DAWSON.

Production of barium peroxide. A. F. MEYERHOFER (G.P. 426,735, 6.11.23. Addn. to 426,034 ; B., 1926, 539).—Barium silicofluoride produced by the treatment of barium phosphate with hydrofluosilicic acid and subsequent separation of phosphoric acid, is decomposed by heat into barium fluoride and silicon tetrafluoride, the latter being used for the production of more hydrofluosilicic acid. The barium fluoride is treated with nitrates of metals which form insoluble fluorides, yielding barium nitrate, which is converted successively into the oxide and peroxide by the usual methods. L. A. COLES.

Electrolytic production of hydrogen peroxide. SIEMENS & HALSKE A.-G. (F.P. 603,043, 10.9.25. Conv., 26.9.24).—Cathodes for use in the electrolytic production of hydrogen peroxide from persulphuric acid or its salts are constructed of gold or of lead, or of aluminium or its alloys coated with gold. L. A. COLES.

Preparing highly concentrated sulphur dioxide gas. S. G. S. DICKER. From K. KUDOH (E.P. 252,928, 17.7.25).—Material containing sulphur is mixed with approximately 10—30% by weight of pyrites cinders and charged into a furnace containing an atmosphere of gaseous sulphur dioxide and at least 22% by vol. of oxygen, together with some sulphur trioxide. The gases leaving the furnace, after cooling, are washed with sulphuric acid and a portion is recirculated. H. ROYAL-DAWSON.

Manufacture of pure sulphur. J. RILEY & SONS, LTD., and W. H. BENTLEY (E.P. 252,938, 17.8.25).—Crude sulphur is dissolved in the mother liquor from a previous operation to form a solution of polysulphide, which is treated with hydrogen sulphide at a low temperature and under pressure to precipitate elemental sulphur. H. ROYAL-DAWSON.

Refining or treating sulphur. W. P. THORNTON (U.S.P. 1,586,539, 1.6.26. Appl., 21.7.23).—Molten sulphur is refined by passing it repeatedly through hot water in a closed vessel. H. ROYAL-DAWSON.

Concentrating nitric acid in a column. W. BÜSCHING (U.S.P. 1,590,494, 29.6.26. Appl., 14.5.23. Conv., 17.8.22).—See G.P. 398,320 ; B., 1924, 942.

Catalyst for synthesis of ammonia. I. W. CEDERBERG, M. FJELLANGER, and V. GRUNER, Assrs. to NORSK HYDRO-ELEKTRISK KVAELSTOF-AKTIESELSKAB (U.S.P. 1,589,682, 22.6.26. Appl., 7.9.21. Conv., 7.9.20).—See E.P. 168,902 ; B., 1923, 95A.

Treating boron-containing minerals., A. KELLY, Assr. to BORAX CONSOLIDATED, LTD. (U.S.P. 1,590,099, 22.6.26. Appl., 2.1.24. Conv., 16.1.23).—See E.P. 208,929 ; B., 1924, 173.

Recovering sulphur dioxide from gases with which it may be associated. A. H. EUSTIS (U.S.P. 1,589,133, 15.6.26. Appl., 7.2.20).—See E.P. 168,627 ; B., 1921, 846 A.

See also pages 649, **Recovering sulphur from gas-purifying material** (G.P. 426,385). 668, **Cement from waste lime** (E.P. 253,448). 673, **Copper from ores etc.** (E.P. 253,370). 683, **Chromium from leather waste** (G.P. 427,807).

VIII.—GLASS; CERAMICS.

Index of refraction of glass at higher temperatures. C. G. PETERS (Sci. Papers U.S. Bur. Standards, 1926, 20, [521], 635—659).—Measure-

ments of the index of refraction of pyrex glass and of 5 crown and 3 flint glasses, over the range 20—700°, indicated that the index increased until the critical expansion region, near 500°, was reached, then decreased in this region, and finally increased again above the softening temperature. In every case the value was much larger than the index as computed from the density relation $(n-1)/d=C$, this increase of the index with rise of temperature being probably due to the same cause which shifts the absorption band towards the longer wavelength region. The method employed was that of placing a plate of the glass in contact with two interferometer mirrors, so that two adjacent sets of interference fringes were visible, one produced by light passing through the glass, the other by light passing through an equal space in vacuum.
A. COUSEN.

Non-actinic cobalt-blue glass. W. W. COBLENTZ and A. N. FINN (J. Amer. Ceram. Soc., 1926, **9**, 423—425).—A glass of the composition, SiO_2 69·5, CaO 6·0, CeO_2 6·8, CoO 0·1, Na_2O 17·6, melts and fines readily at 1425° and works easily. The apparent colour is very similar to that of the corresponding soda-lime cobalt glass, but the absorption of the ultra-violet rays is greater, being practically complete with a glass 5 mm. thick. A. COUSEN.

Attack of arsenic compounds on fireclay refractory material. W. E. S. TURNER (J. Amer. Ceram. Soc., 1926, **9**, 412—417).—The conclusion of McSwiney (B., 1925, 546) that arsenious oxide employed in the glass batch was responsible for the rapid corrosion of the tank blocks, was examined in the light of analyses of several series of soda-lime glasses, made from batches with varying proportions of arsenious oxide, but with the other batch constituents, as well as the time and temperature of melting, constant in each series. Results, based on the amount of iron oxide and alumina in the glass, indicated that even when amounts of arsenious oxide 4 or 5 times those used in practice were employed, no marked corrosion took place at 1400°, and the attack at 1450° and 1500° was only marked with amounts of arsenious oxide in excess of that used in practice. A. COUSEN.

Anomalous flocculation of clay. KERMACK and WILLIAMSON.—See A., July, 679.

PATENTS.

Manufacture of pottery or the like. J. W. MELLOR (E.P. 253,184, 9.3.25).—The composition of the body is adjusted so that the body matures at the same temperature as the glaze. The biscuit firing can then be eliminated and the whole article fired in the glost kiln. B. W. CLARKE.

Production of refractory oxide ware. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,585,779, 25.5.26. Appl., 8.3.22).—A mixture of thoria, cryolite, phosphoric acid, and water is shaped and then fired. B. W. CLARKE.

Ceramic and refractory making process and material. A. G. BETTS (U.S.P. 1,585,826, 25.5.26. Appl., 3.3.23).—Impurities such as iron in otherwise good quality clays and refractories are removed as volatile chlorides during the burning process, if the raw material be mixed with aluminium chloride solution instead of with water. E. S. KREIS.

Refractory and ceramic process and materials. A. G. BETTS (U.S.P. 1,585,827, 25.5.26. Appl., 12.3.23).—Ceramic and refractory materials which are resistant to acids are made up with a solution of basic aluminium chloride instead of with pure water. Besides causing the removal of impurities such as iron, as volatile chlorides, during the firing, the basic aluminium chloride solution possesses advantages as a bonding material because it is a non-crystallising solution and is equivalent to a colloidal solution of hydrated aluminium oxide. The pores of the resulting material are filled up with aluminous material which increases the density and diminishes the shrinkage on vitrifying.
E. S. KREIS.

Composition of matter. [Utilisation of scrap fused silica.] W. W. WINSHIP, Assr. to THERMAL SYND., LTD. (U.S.P. 1,587,057, 1.6.26. Appl., 8.5.25).—Fused silica scrap and quartz are ground and mixed to a stiff paste with sodium silicate solution, preferably of *d* 1·345. After being moulded, the articles are dried for several days and then treated with concentrated hydrochloric acid. They are then washed, or they may be treated with ammonia previous to washing.
E. S. KREIS.

Drying clays and similar materials. T. H. RHOADS, Assr. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,587,419, 1.6.26. Appl., 21.6.21).—Ceramic articles are introduced into a drying compartment provided with spraying devices; the wet bulb temperature of the compartment is raised almost to that of the article, which is then dried gradually by raising the dry bulb temperature. A flow of moisture from the interior of the article to the exterior is then induced by bringing the air in the compartment almost to the dew point, and finally by progressively raising the wet and dry bulb temperatures of the air the article is brought to the required degree of dryness. B. W. CLARKE.

Production of fireproof and acid-proof material. DEUTS. GASGLÜHLICHT-AUER-GES. M.B.H. (F.P. 602,134, 17.8.25. Conv., 13.8.24).—The material is prepared from one or more hafnium compounds, preferably in admixture with other refractory material, such as silica or magnesia.
L. A. COLES.

Making vitreous silica. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of P. K. DEVERS (E.P. 240,489, 28.9.25. Conv., 27.9.24).—See U.S.P. 1,536,821; B., 1925, 548.

Treatment of clay. W. FELDENHEIMER (U.S.P. 1,588,956, 15.6.26. Appl., 16.6.25. Conv., 7.8.24).—See E.P. 242,357; B., 1926, 55.

Tunnel kiln. A. McD. DUCKHAM (U.S.P. 1,590,154, 22.6.26. Appl., 4.11.24. Conv., 12.11.23).—See E.P. 228,987; B., 1925, 318.

Sheet glass drawing apparatus. LIBBEY-OWENS SHEET GLASS Co. (E.P. 240,448, 18.9.25. Conv., 24.9.24).

Manufacturing plate glass. F. RIECHERS (E.P. 251,632, 28.4.26. Conv., 4.5.25).

Glass-making furnaces. J. and A. BOUCHER (E.P. 243,322, 6.10.25. Conv., 22.11.24).

Apparatus for comminuting or grinding china clay. R. G. VARCOE and H. P. GARVEY (E.P. 253,694, 18.5.25).

IX.—BUILDING MATERIALS.

Determination of soluble silica in cements, mortars, and concretes. D. FLORENTIN (Compt. rend., 1926, **183**, 53—55).—The cement (1 g.) or mortar (4 g.) is added, little by little, to 50—60 c.c. of cold hydrochloric acid (d^{15} 1·12). The reaction vessel should be cooled in a current of cold water. The solution is filtered and the silica in the filtrate rendered insoluble in the usual manner. If a small quantity of the soluble silica is precipitated the residue is treated with a warm 5—6% sodium carbonate solution, this extract being added to the main filtrate. L. F. GILBERT.

Cement–lime mortars. H. V. JOHNSON (Tech. Papers U.S. Bur. Standards, 1926, **20**, [308], 241—274).—The addition of lime to cement mortars increases the amount of water required to bring the mix to a given consistency, and therefore increases the shrinkage of the mortar. The addition of lime increases the strength of cement mortars leaner than 1 : 2 by weight, and also increases the density of the mortar. The addition of cement to a lime plaster or mortar increases the strength and shortens the time of setting without affecting the plasticity or the ease with which the mass can be worked. B. W. CLARKE.

Strengthening and indurating concrete with sulphur. W. H. KOBBÉ (Eng. News-Rec., 1926, **96**, 940—942).—Impregnation of pre-cast concrete with sulphur results in an increase in strength and a decrease in absorption. The concrete products, preferably well cured, are immersed in a bath of molten sulphur, usually kept at a temperature of 130—150°, until the requisite degree of absorption is obtained. The treatment is applicable to all types of concrete and cement mortars including high-alumina cement and with any aggregate chemically inert toward sulphur. Concrete absorbs ordinarily from 10 to 25% of sulphur, depending upon the character of the mix and its porosity. The rate of absorption of the molten sulphur is dependent upon a number of factors, moisture content probably being the dominant one. Usual mixes, if fairly dry, may be impregnated to a depth of at least 2 in. in 8 hrs. Standard tensile briquettes of cement mortars, which ordinarily break at about 150 lb., showed increases in strength to 1000—1700 lb. and occasionally withstood a pull of 2000 lb. per sq. in. after impregnation. Under compression concrete impregnated with sulphur showed an increase in strength over the untreated similar to that indicated in the case of the tensile tests. Water absorption is reduced to less than 2—3%. The cost of treatment is comparable with the cost of creosoting timber. Electrolytic cells made of concrete impregnated with sulphur withstand successfully the very corrosive action of a mixture of hot ferrous and ferric chloride solution. W. T. LOCKETT.

Indurating wood with sulphur. W. H. KOBBÉ (Chem. Met. Eng., 1926, **33**, 354—356).—Soft wood immersed in molten sulphur for about 10 hrs. at 120—150° absorbs up to 70% by weight of sulphur, whereby the strength and hardness of the wood are considerably increased. The sulphur acts as a preservative owing to the complete sealing of the pores. A similar hardening effect occurs with hardwoods for use as paving blocks, sleepers, etc., on treatment with molten sulphur. B. W. CLARKE.

System calcium oxide–ferric oxide–silica. HANSEN and BOGUE. See A., July, 684.

PATENTS.

Cement and lime burning. G. E. HEYL (E.P. 252,780, 3.3.25).—A dry powdered mixture of cement- or lime-forming materials is projected into a region of a combustion chamber maintained at a vitrifying temperature by a flaming jet of oil or powdered fuel, in such proportions that the ash of the fuel is negligible in comparison with the amount of cement or lime produced. B. W. CLARKE.

Manufacturing process for cements containing iron and alumina combinations. E. MARTIN (U.S.P. 1,586,099, 25.5.26. Appl., 11.9.24).—A mixture of raw materials containing calcium carbonate, ferric oxide, and alumina in the proportion of more than 2 mols. $CaCO_3$ for each mol. of Fe_2O_3 and 1 mol. $CaCO_3$ for each mol. of Al_2O_3 is burnt at a temperature below the m.p. of any of the ingredients. B. W. CLARKE.

Manufacture of cement out of spent or waste lime. R. ILLEMANN (E.P. 253,448, 31.12.25).—Waste lime from the Leblanc soda process is heated in a revolving furnace at a temperature up to 700°, according to the properties required in the finished product, and after grinding, mixed with small quantities of alum, potash, or borax (which may be added before the heating if desired), or with ground glue. The product forms a hard cement or plaster on mixing with water. B. W. CLARKE.

Mineralising fibrous materials. NOVOCRETES, LTD., and G. O. CASE (E.P. 252,906, 27.3 and 27.9.24. Addn. to 225,912; cf. B., 1925, 134).—Fibrous material, *e.g.*, sawdust, is damped to saturation with a solution of a metal salt, *e.g.*, a chloride, and mixed with a solution which will produce an insoluble precipitate in or on the fibrous material, without

initiating setting, thus forming a granular material suitable for use with cement, concrete, and the like.
B. W. CLARKE.

Manufacture of light forms of concrete or artificial wood composed of loose or fibrous organic materials and cement, concrete, or the like. BROADWAY TRUST CO., LTD., C. D. BURNEY, and J. E. TEMPLE (E.P. 253,007, 16.12.24).—Organic material, *e.g.*, sawdust, is heated gradually to 120—130°, or treated with materials such as oils, resins, soaps, emulsions, etc., in order to render it water-repellent and less susceptible to change in volume with change of moisture content. It is then mineralised (cf. E.P. 244,178 ; B., 1926, 130) to make it suitable for combining with cement etc. Alternatively the material may be treated with a weak solution of an alkali or an acid, or with a solution which will dissolve resinous compounds, before mineralising and mixing with cement.
B. W. CLARKE.

Preserving wood by impregnation. GEBR. HIMMELSBACH A.-G. (E.P. 253,041, 23.2.26. Conv., 30.11.25).—Wood is steamed strongly at a high temperature and then dried briskly at about 150°. A large number of cracks are thereby produced in the surface of the wood, resulting in a more complete and uniform impregnation when the timber is treated in the usual way with preserving solutions. Cresol etc. may be used with the steam if desired.
B. W. CLARKE.

Emulsification of tar, bitumen, etc. (E.P. 252,449).—See II.

Preparation of Trinidad pitch lake asphalt, etc. (E.P. 252,802).—See II.

Blown oil asphalts (U.S.P. 1,586,376).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Phosphorus in wrought iron. H. S. RAWDON and G. EPSTEIN (Amer. Iron and Steel Inst., Mar., 1926; Engineering, 1926, **172**, 13).—Comparative tests on wrought iron made from the same cast of pig iron by hand and by mechanical puddling indicated that there was no reason why the properties of mechanically puddled iron should be inferior to those of the hand puddled product. Phosphorus was eliminated more readily than by hand work, though non-uniformity of distribution persisted, and in view of this the average phosphorus content should be considerably less than 0·18%.
C. A. KING.

Malleable cast iron and the method of its graphitisation. T. KIKUTA (Sci. Rep. Tohoku, 1926, **15**, 115—155).—The graphitisation of white cast iron has been studied using the dilatometer. To obtain black heart cast iron the graphitisation is best performed in two stages. For the complete graphitisation of free cementite the article must be heated at a high temperature above the Ar1 point, and then to decompose the eutectoid cementite prolonged heating at a temperature below, or very slow cooling from the Ar1 point is essential. The annealing temperature greatly affects the graphitisation in both the first and second stages; the time required to complete them decreases logarithmically with rise of temperature. Thickness of casting increases the size of the free cementite and this consequently becomes more difficult to graphitise; moreover, the higher the tapping temperature of the melt the harder graphitisation becomes. Increase in silicon content causes a logarithmic decrease in the time of both the first and second stages. Carbon is more effective than silicon in the second stage, but it scarcely affects the first stage. Manganese lessens graphitisation, the effect being particularly great in the second stage, so that not more than 0·5% should be present in black-heart malleable iron castings. Sulphur behaves similarly, and the sulphur content should be limited to 0·6%. Phosphorus assists graphitisation in the first stage, but for the second stage the amount should not exceed 0·3%. Rapid heat treatment of a sample of white cast iron containing more than 2·5% of carbon and 1% of silicon gave a black-heart malleable iron. Black-heart malleable cast iron grows with repeated heating and cooling through the A1 range in a similar manner to grey cast iron. The mechanism of graphitisation of white cast iron is explained as due to the catalytic action of free carbon oxides present in the casting; a nucleus of temper carbon is formed at the boundary of the cementite and graphitisation is accelerated by the affinity between iron and the included elements.
M. CARLTON.

Diffusion of carbon in α-iron. E. ZINGG (Stahl u. Eisen, 1926, **46**, 776—777).—Experiments were made to test whether carbon absorption takes place below Ac3 or Ac2. Cementation was carried out in a mixture of hydrogen, hydrocarbons, and carbon monoxide obtained by the removal of carbon dioxide, water vapour, hydrogen sulphide, ammonia and other nitrogen compounds from illuminating gas. The materials used were Swedish wrought iron 0·1 mm. and 0·05 mm. thick and a steel containing 1·2% C, 0·23% Si, and 0·27 Mn, 1 mm. thick, which were heated at 780—800° and 670—690° respectively for 20 hrs. and also for 60 hrs. The specimens were cooled in the gas and afterwards etched with sodium picrate solution. Microscopical examination showed that from 650° upwards to 800° the surface layers of the test-pieces were converted into cementite. In the α-iron region sharp boundaries were formed between the cementite layers and the ferrite grains, but the cementite also penetrated into the interior along the crystal boundaries. Inside the ferrite grains small spheres like granular cementite appeared. In the mixed-crystal region the boundaries of the cementite layers were also sharp. When sufficient time was allowed the steel was converted almost completely into cementite. Fry's distinction (cf. B., 1923, 932 A) between pure and reaction diffusion was confirmed. The former only takes place up to

the content of the saturated mixed-crystals, whilst with reaction diffusion the carbon content of the highest chemical compound can be attained.
T. H. BURNHAM.

Data relating to basic open-hearth steel practice. A. N. DIEHL (Amer. Iron and Steel Inst., May, 1926; Engineering, 1926, 172, 13).—Complete records of the operation of basic steel furnaces of 50—75 tons capacity were kept. When molten pig-iron alone was charged 98% of the silicon content was eliminated within 1 hr., and manganese was oxidised almost as quickly, with, however, a marked reversion from slag to the metal in the later stages of the heat. Phosphorus was oxidised in the first stages and the duration of the heat was due to the time required for the elimination of carbon. When a carbon content of more than 0·25% was desired re-carburising was effected best by the addition of molten pig-iron, though for relatively low-carbon steels anthracite was added as the metal ran into the ladle. Manganese was added in the ladle or larger quantities in the furnaces and ferrochromium also in the furnace. Nickel was introduced as scrap or pig into the furnace, only the final adjustment being made before pouring after a preliminary analysis. C. A. KING.

Absorption of nitrogen by iron in the basic open-hearth process. E. H. SCHULZ and R. FRERICH (Mitt. Versuchsanst. Deutsch-Luxemburg. Bergwerks u. Hütten-A.-G. Dortmunder Union, 1925, 1, 251—257; Chem. Zentr., 1926, I., 3427).—The amount of nitrogen taken up by steel in the basic open-hearth process is greater the higher the temperature, the longer the time of blowing, and the larger the volume of air passed through the metal. These factors are especially pronounced during the dephosphorising period, particularly the time factor, which has a far greater influence on the nitrogen absorption than any of the others.
A. R. POWELL.

Equilibrium between austenite and the carbon oxides. G. TAKAHASHI (Sci. Rep. Tohoku, 1926, 15, 157—175).—The concentration of carbon monoxide in the gas mixture ($CO+CO_2$) must increase with rise of temperature in order to maintain austenite at the same carbon content; if, however, the carbon monoxide concentration be increased beyond a certain limit, the carbon content in austenite increases and free cementite is produced; diminishing the carbon monoxide concentration increases the percentage of pure iron. If a specimen be heated above 900° and the carbon monoxide in the gas phase be less than a certain limiting value, conversion into ferrous oxide occurs. The carbon content in austenite in equilibrium with free cementite increases with rise of temperature, whilst if austenite be in equilibrium with α-iron or ferrous oxide, the carbon content decreases with temperature rise. The carbon monoxide content in a gas mixture in equilibrium with austenite, austenite and free cementite, or austenite and ferrous oxide increases with rise in temperature but decreases for austenite and α-iron. Cementation is not produced by a gas mixture of which the carbon monoxide concentration is less than that in equilibrium with austenite, α-iron, or ferrous oxide. Curves are given from which may be deduced the conditions under which carburisation etc. may take place. The A1 transformation point has been determined to be 726° by cementation in carbon monoxide gas.
M. CARLTON.

Cementation of steel by silicon. L. GUILLET (Compt. rend., 1926, 182, 1588—1589; cf. Fry, B., 1923, 932 A).—In presence of ammonium chloride cementation of steel by ferrosilicon (75—80% Si) takes place extremely rapidly at temperatures in the region 1100—1200°. The surface layer contains about 15% Si and is very brittle. J. S. CARTER.

Electrolytic iron from ilmenite ores. R. H. MONK and R. J. TRAILL (Canad. Chem. Met., 1926, 10, 137—139).—Experiments were made on a Quebec ore containing 31·6% TiO_2 and 40·5% Fe. Three parts of crushed ilmenite were mixed with one part of charcoal and heated in a crucible to 900—950° for 1½ hrs. After running over a Wetherill magnetic separator the sponge product contained 40·5% of metallic iron, 46·7% of total iron, 36·9% TiO_2, and 2·0% C. It was treated with a leaching liquor consisting of a mixture of ferrous and ferric chlorides, the charge of sponge being adjusted to cause complete reduction of all the iron to the ferrous state. A combination of thickening and settling is recommended for separating the insoluble residue. The ore tested contained small amounts of copper, lead, nickel, and cobalt which were removed by means of calcium sulphide before the electrodeposition of the iron. Nickel, copper, and cobalt below 0·02 and lead below 0·002 g. per litre had no ill effect. For the deposition of the iron a diaphragm cell of 12 litres electrolyte capacity was used, having as cathode a steel mandrel rotating at 250—400 r.p.m. The current density was 100 amp. per sq. ft. Satisfactory deposition was obtained only at 80—95°. The titanium oxide content of the ilmenite can be recovered by any of the well-known processes. The authors consider the process can be made commercially successful, starting with the production of sponge iron in a rotary furnace on the lines of the direct-fired rotary kiln described in the U.S. Bureau of Mines report No. 2656. T. H. BURNHAM.

Hydrometallurgical treatment of iron sulphide ores for the production of electrolytic iron and the recovery of sulphur and other metals as by-products. R. J. TRAILL and W. R. MCCLELLAND (Canad. Dept. Mines, 1926, [643], 92—102).—Pyrrhotite dissolves fairly readily in ferric chloride solution yielding ferrous chloride and free sulphur. Pyrites is insoluble in this reagent, but, by passing it through a furnace kept at 750—800°, it is reduced to ferrous sulphide with the liberation of half the sulphur, 50—65% of which can easily be recovered on a commercial scale, and the porous ferrous sulphide obtained dissolves readily in ferric chloride. Solution is effected at 95° with spent electrolyte containing

about 7% each of ferric and ferrous chlorides. The reduced solution is filtered and boiled with sponge iron to remove copper and the bulk of the lead. No efficient process has yet been evolved to remove the zinc, so that in the subsequent electrolysis this is deposited with the iron and can be eliminated only by melting. A. R. POWELL.

Combined pyro- and hydro-metallurgical process for the treatment of nickeliferous pyrrhotite ores with the subsequent recovery of iron, sulphur, nickel, and copper. H. C. MABEE and A. E. SMAILL (Canad. Dept. Mines, 1926, [643], 105—108).—The present processes for recovering nickel and copper from Sudbury pyrrhotites waste the whole of the iron and sulphur content. If the ore is smelted in a reverberatory or electric furnace with limestone to flux all the gangue a low-grade nickel–copper matte is obtained which contains practically the whole of the nickel, copper, iron, and sulphur originally present. By roasting the matte at 450—500° 85% of the sulphur is recovered as sulphur dioxide. The roasted material is then mixed with 10% of sodium chloride and 5—10% of water and introduced into a rotating furnace held at 450—500° in such a way that the charge is gradually heated as it passes through the furnace. Leaching of the product results in solution of 98% of the copper and 75% of the nickel, leaving an iron oxide containing 3—4% Ni and about 0·4% S which could be used for the direct production of nickel steel. A. R. POWELL.

Specific heat of iron–nickel alloys. M. KAWAKAMI (Sci. Rept. Tohoku, 1926, **15**, 251—262).—Alloys of iron and nickel of varying composition were subjected to different heat treatments, *i.e.*, annealed, annealed and then cooled in liquid air, annealed and quenched in water, and their mean specific heat between 30° and 300° determined by the method of mixtures. The specific heat varies somewhat with different structures, *e.g.*, the specific heat of martensitic alloys is less than that of nickel-ferrite and this in turn is greater than that of austenitic alloys. The maximum specific heat of nickel–iron alloys is connected with the minimum expansibility in accordance with Grüneisen's law (Ann. Physik, 1908, [iv.], **26**, 211). M. CARLTON.

Determinntion of small amounts of bismuth in copper. C. O. JONES and E. C. FROST (Ind. Eng. Chem., 1926, **18**, 596).—Minute proportions of bismuth in copper (about 0·002%) were most accurately determined colorimetrically by precipitating with sodium phosphate and ammonium carbonate from a nitric acid solution of the copper after adding a crystal of ferric sulphate and making the solution ammoniacal. The precipitate is dissolved in dilute sulphuric acid, hydrogen sulphide passed through, and the antimony and arsenic sulphides are separated from the bismuth sulphide by potassium hydroxide or ammonium sulphide. The remaining copper is separated by precipitating the bismuth as sulphide in ammoniacal potassium cyanide solution, and the bismuth sulphide is then dissolved in nitric acid, the solution evaporated with sulphuric acid, and any lead sulphate removed. After addition of potassium iodide and sulphurous acid the colour is compared with that of standard solutions similarly treated. D. G. HEWER.

Equilibrium diagram of copper–tin system. T. ISIHARA (Sci. Rep. Tohoku, 1926, **15**, 225—246).—The equilibrium diagram of the copper–tin system previously described (A., 1925, ii., 122) is confirmed by results of dilatometric and hardness measurements. Dilatometric measurements show that the change $\alpha+\delta\rightarrow\beta$ appears as a contraction while $\eta+\delta\rightarrow\gamma$ produces an expansion. The hardness of specimens measured by the Shore and Brinell methods and the Honda–Sato dynamic method give results in fair agreement. The eutectoid transformation $\alpha+\delta\rightarrow\beta$ is accompanied by an increase and the change $\delta+\eta\rightarrow\gamma$ by a decrease in hardness. The relation between hardness and composition in specimens quenched at different temperatures agrees with the equilibrium diagram, as also do microscopical observations. M. CARLTON.

Formation of carbon tetrafluoride in the technical recovery of aluminium. W. D. TREADWELL and A. KÖHL (Helv. Chim. Acta., 1926, **9**, 681—691).—No trace of carbon tetrafluoride is produced in the electrolytic production of aluminium from alumina–cryolite baths although from theoretical considerations it should be formed at the anode as readily as carbon dioxide. This is probably due to the homopolar nature of carbon tetrafluoride so that in the presence of aluminium oxide the oxygen is preferably discharged at the anode. In support of this is the fact that formation of carbon tetrafluoride during the electrolysis of cryolite in a mangnesia crucible ceases as soon as a trace of oxide has dissolved from the surface of the crucible. Carbon tetrafluoride dissolves in water to the extent of 7·45 c.c. per 100 c.c. at 16° and 6·5 c.c. per 100 c.c. at 20°. The solution is stable and is not hydrolysed by boiling with aqueous alkalis, although alcoholic potassium hydroxide causes slow hydrolysis. On heating to redness carbon tetrafluoride is decomposed with the formation of a white solid containing carbon and fluorine and with the liberation of hydrogen fluoride, the etching properties of which serve as a test for the presence of the tetrafluoride. A. R. POWELL.

Corrosion of aluminium by water. I. L. W. HAASE (Z. Elektrochem., 1926, **32**, 286—289).—Experiments on the corrosion of aluminium by water have been made by placing samples of surface, drinking, and sewage water in corked aluminium flasks and following the changes in the hydrogen-ion concentration and the conductivity and determining the aluminium hydroxide formed. All the samples of water attacked the aluminium more or less, sometimes with formation of holes. The corrosion was greatest at the bottom of the flasks and at the liquid–air boundary. This may be explained both electrolytically and electrochemically. The aluminium hydroxide was in some cases partly colloidal. Both acid and alkaline waters attack aluminium owing to its amphoteric nature and

suffer a corresponding change in their p_H values, but the final stage is never neutral since a small part of the hydroxide is always dissociated in a way depending on whether hydrogen or hydroxyl ions preponderate. N. H. HARTSHORNE.

Equilibrium diagram of the aluminium–zinc system. T. ISIHARA (Sci. Rep. Tohoku, 1926, **15**, 209—224; cf. B., 1925, 246).—Eutectoid transformation in aluminium–zinc alloys has been studied using the dilatometer. Alloys containing 1—60% of aluminium show a discontinuous expansion at 280°, the magnitude of which increases up to 21% Al and is directly proportional to the amount of the eutectoid present. The eutectoid change $\alpha+\gamma\rightarrow\beta$ is accompanied by a discontinuous increase in the scleroscope (Shore) hardness. The hardness of quenched specimens reaches a maximum in less than 1 hr. after quenching and then decreases rapidly, finally becoming asymptotic. This ageing effect is assumed to be chiefly due to an intermediate β^1 form: $\beta+\beta^1\rightarrow\alpha+\gamma$, but no difference between β and β^1 can be found either microscopically or by *X*-ray analysis. M. CARLTON.

Boron in aluminium and its alloys. P. HAENNI (Rev. Mét., 1926, 23, 342—352).—A fuller description and amplification of earlier work (cf. B., 1926, 58). A small quantity of boron improves the structure and properties of copper–aluminium and zinc–aluminium alloys; it also improves slightly the resistance of the alloys to corrosion. A. R. POWELL.

Influence of the thermal zone of work on the selection of metals for aviation motors. Application to exhaust valves. GRARD (Rev. Mét., 1926, **23**, 317—330).—Fuller details are given of work the results of which have already been published (cf. B., 1926, 92). A. R. POWELL.

Hardening of printing type alloys. A. TRAVERS and HOUOT (Compt. rend., 1926, **182**, 1627—1628).—The alloys as cast show a considerable contraction during heating for dilatometric examination; the magnitude of the contraction under comparable conditions increases with the tin content. The contraction also varies with the casting temperature, increasing 20% for example when the casting temperature is raised from 300° to 360°. It is diminished by previous annealing of the alloy. Annealing occurs even at the ordinary temperature, but is not complete even after 6 weeks; it is completed in 24 hrs. at about 200°. The alloys become exceedingly brittle after ageing for several weeks at the ordinary temperature. J. S. CARTER.

Determination of fluorine [in ores]. F. G. HAWLEY (Ind. Eng. Chem., 1926, **18**, 573—576).—The fluorine is determined indirectly from the chlorine contained in precipitated lead chlorofluoride. The sample is adjusted to contain at least four times as much silica as fluorine, and fused with sodium and potassium carbonates. After adding just enough sodium peroxide for oxidation of any sulphur present (which is liable to cause loss of fluorine), the fused mass is heated with water until disintegrated, the residue boiled again with water with addition of sodium carbonate, filtered, concentrated hydrochloric acid added to the filtrate, the liquid warmed, neutralisation completed with nitric acid, and glacial acetic acid and 10% lead acetate solution containing 1% of acetic acid added to the slightly acid solution. The precipitated lead chlorofluoride is filtered off, washed, redissolved in nitric acid, chlorine precipitated by silver nitrate, and determined. The presence of lead and zinc have little effect on the results. The method gives the best results for low- to medium-grade ores. D. G. HEWER.

Reports of investigations. Ore dressing and metallurgical laboratory. W. B. TIMMS, C. S. PARSONS, R. K. CARNOCHAN, and J. S. GODARD (Canad. Dept. Mines, 1926, [643], 8—91).—Details with flow-sheets and analyses of the products are given of numerous concentration tests on zinc–lead, gold–copper, molybdenite, silver–lead, garnet, and titanite ores, of cyanide tests on gold ores, and of cleaning and trimming experiments on the product of a mica mine. A. R. POWELL.

Concentration of Lake George antimony ores. C. S. PARSONS (Canad. Dept. Mines, 1926, [643], 110—115).—The ore consists of stibnite with a small amount of mispickel disseminated in fissures in slate and quartzite. It averages 11·6% Sb and 0·4% As. Flotation tests on the ore passing through 65-mesh gave a concentrate containing 64% Sb and 0·28% As with a recovery of 86% of the antimony; the oil used consisted of a mixture of 40% of coal tar and 60% of coal tar creosote with a small addition of pine oil, and the pulp was neutral. An acid pulp gave an extraction of 95% in a concentrate assaying 63·4% Sb and 0·31% As, whereas an alkaline pulp gave a poorer extraction and over 1% As was present in the concentrate. A. R. POWELL.

Concentration of lead–zinc ores of Eastern Canada. C. S. PARSONS (Canad. Dept. Mines, 1926, [643], 109—110).—An historical summary. A. R. POWELL.

Recovery of sulphur from waste gases from metallurgical furnaces. E. WILL (Mitt. Versuchsanst. Deutsch-Luxemburg. Bergwerks- u. Hütten-A.-G. Dortmunder Union, 1925, **1**, 237—242; Chem. Zentr., 1926, I., 3426).—Attempts to recover the sulphur dioxide from waste gases from furnaces by passing the gases at 450—800° over ferric oxide, copper chromate, or chromic oxide and alumina contact masses failed owing to the large proportion of moisture present causing the formation of sulphates. By cooling the gases to 10°, however, the mist formed by the condensation of this moisture resulted in a precipitation of 50% of the sulphur dioxide content of the gases. A. R. POWELL.

A2 line in the equilibrium diagram of the iron–carbon system. K. HONDA (Sci. Rep. Tohoku, 1926, **15**, 247—250).—See B., 1926, 194.

See also A., July, 665, **Magnetic properties of single crystals of iron** (HONDA, KAYA, and

Masuyama). 666, **Behaviour of single crystals of aluminium under static and repeated stress** (Gough, Hanson, and Wright); **Influence of mean principal stress on flow of iron, copper, and nickel** (Lode); **Properties of gold–silver–copper alloys** (Sterner-Rainer). 669, **Dilatometric investigation of A3 and A4 transformations in pure iron** (Sato). 671, **Hardness of copper–tin alloys** (Mallock). 673, **Absorption of hydrogen and carbon dioxide by pyrophoric iron, nickel, and cobalt** (Nikitin). 683, **Ternary diagram of system iron–carbon–copper** (Ishiwara, Yonekura, and Ishigaki). 684, **Equilibrium between carbon monoxide, carbon, and carbon dioxide, and reactions between ferrous oxide and carbon, and between carbon monoxide and iron** (Falcke and Fischer). 692, **Tarnishing of copper in sulphur vapour** (Fischbeck); **High-temperature oxidation of metals; Low-temperature oxidation of metals** (Dunn). 699, **Preparation of pure zirconium** (de Boer and Fast). 705, **Detection of tin in minerals, using the blowpipe** (Braly). 707, **Oberhoffer's etching mixture** (Heinrich and Voigt).

Equipment for high-pressure reactions. Ernst.—See I.

Coke testing. Haven.—See II.

Electrodes of V2A-steel in electro-analysis. Schleicher and Toussaint.—See XI.

Prevention of corrosion. Baylis.—See XXIII.

Patents.

Kiln for production of iron sponge. S. E. Sieurin (E.P. 252,899, 4.6.25).—In a kiln for the production of iron sponge the containers for the charge are in the form of vertical polygonal retorts, built up of shaped bricks so arranged that horizontal heating ducts are formed between the retorts. In one form of construction the bricks are made with flanges to space the retorts and so form ducts. Certain of the vertical retorts may be used as combustion chambers, having side outlets into the heating ducts. C. A. King.

Production of metals [iron, chromium, and manganese] and their alloys [from ores]. S. Heuland (F.P. 602,448, 22.8.25).—Iron, chromium, or manganese ores or mixtures of these are melted in an electric furnace with a reducing agent sufficient to produce only a small amount of metal which will contain all the deleterious impurities in the ores, *e.g.*, phosphorus, carbon, or iron. The remainder of the metal is then reduced from the fused slag by addition of calcium silicide. A. R. Powell.

Recovery of metals, especially iron and steel [from ores and scrap]. L. Blanchet (F.P. 602,703, 10.12.24).—Iron ores or scrap iron are heated to 400—1200°, preferably 600—1200°, and a stream of hydrocarbon vapours is passed over them whereby finely divided carbon is deposited on the ore or scrap and lighter oils are recovered from the issuing gases together with inflammable gas which is used for heating the furnace. The carbon-coated ore or scrap is subsequently smelted in a separate furnace. A. R. Powell.

[Non-oxidising iron–nickel] alloys. R. L. Spitzley and A. M. Thompson, Assrs. to Alloys Foundry Co. (U.S.P. 1,587,992–4, 8.6.26. Appl., [a] 22.5, [b] 25.5, and [c] 2.6.25).—Non-oxidising alloys suitable for fusing with cast-in steel parts contain (a) 56% Ni, 8% Cr, 7% Mn, 26% Fe, 2% Si, and 1% W, (b) 64% Ni, 7% Mn, 14% Fe, 2% Si, 1% W, and 12% Cu, and (c) 58% Ni, 8% Cr, 7% Mn, 14% Fe, 1% W, and 12% Cu. A. R. Powell.

Casting having silicon-alloy surface. C. B. Jacobs, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,588,111, 8.6.26. Appl., 30.4.21).—A casting of iron which has a surface highly resistant to the action of acids is produced by coating the surface of the mould with a mixture of ferrosilicon particles (15- to 40-mesh) and a suitable binder, hardening the coating, and then casting molten iron at such a temperature that the ferrosilicon is dissolved in the surface layer of metal. C. A. King.

Treatment of solid materials with liquids. [Precipitation of copper with scrap iron.] A. Ramén (E.P. 235,902, 18.6.25. Conv., 20.6.24).—In the precipitation of copper from copper lyes by means of scrap iron, atmospheric oxidation of the copper is prevented by the use of an elongated container rotating or rocking about its inclined longitudinal axis, and maintaining a permanent liquid seal at each end during the continuous passage of the liquid through the container. The outlet is provided with a sieve to retain the scrap iron. The copper is collected in a filter-press, and the liquid leaving the press is, if not exhausted, re-circulated through the container. H. Holmes.

Extraction of copper from ores, concentrates, and residues. H. S. Mackay (E.P. 253,370, 2.7.25).—The ore is roasted in such a way that the whole of the copper and a small part of the iron are converted into sulphates. The product is then leached with dilute sulphuric acid, previously used to finish the leaching of a similar charge, so as to obtain a neutral solution which is then purified by treatment with calcium carbonate (cf. E.P. 238,962, B., 1925, 854). The filtrate is evaporated to obtain crystals of copper sulphate. A. R. Powell.

Electrolytic precipitation of copper. F. F. Frick and C. E. Carstens, Assrs. to Anaconda Copper Mining Co. (U.S.P. 1,586,035, 25.5.26. Appl., 23.10.25).—Waste solutions containing copper obtained during the leaching of copper ores are treated with a basic substance to precipitate the copper, and the resulting sludge is leached with acid to give a copper solution from which the metal is deposited electrolytically. A. R. Powell.

Obtaining electrolytic deposits of chromium. G. Le Bris (E.P. 243,667, 25.5.25.

Conv., 1.12.24).—A bath for the electrodeposition of chromium comprises a colloidal solution obtained by boiling chromic hydroxide with chromic acid in the molecular ratio of 1 : 4 and adding a small amount of sodium perborate or other oxidising salt. Deposition is effected at 4—6 volts with a current density of 7—8 amp./dm.^{2} using lead anodes.

A. R. POWELL.

Apparatus for roasting or sintering ores and the like. METALLBANK & METALLURGISCHE GES. A.-G. (E.P. 244,126, 7.12.25. Conv., 6.12.24).—In a roasting furnace of the circular Dwight and Lloyd type ore is introduced into a number of separate compartments resting on the periphery of the rotating table. Each compartment is connected by a pipe with a segment of a central chamber which rotates over a stationary chamber also divided into segments. Suction in the compartments of the fixed chamber may be regulated by valves as desired, *e.g.*, as the ignited ore passes over the different segments in turn, the draught may be increased gradually.

C. A. KING.

Ore-reducing furnace. W. WINKELMAN (U.S.P. 1,588,217, 8.6.26. Appl., 27.6.23).—In a furnace for reducing ores, inverted troughs are supported on shelves projecting from the side walls of the furnace. Means are provided for producing carbon dioxide, for reducing it to a mixture containing carbon monoxide, and for circulating this mixture through the transverse passages formed by the inverted troughs, in contact with a subdivided ore.

C. A. KING.

Manufacture of metals and alloys. D. CROESE (E.P. 252,455, 26.2.25).—Titaniferous iron sand is caused to fall by gravity through a vertical electric furnace so arranged as to allow an unobstructed path for the falling material. The furnace is heated by a vertical series of arcs, the electrodes of any arc being set at an angle to the electrodes of the adjoining arc. Molten metal (titanium-steel) and slag are collected in a crucible at the bottom of the furnace.

C. A. KING.

Melting and re-melting of metals and metallic waste. K. SCHMIDT (E.P. 253,342, 24.3.25).—The walls of the furnace are directly heated and are then caused to come under the metal to be melted. For example, a reverberatory furnace rotating continuously about an axis may be used, each of the furnace walls being successively heated, brought under the metal to be melted, removed from that position, and reheated. A protective covering is maintained over the metal bath. C. A. KING.

Electrolytic production of metals. E. DUHME, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,567,791, 29.12.25. Appl., 1.11.24).—A small auxiliary cathode is placed near the anode and supplied with alternating current and with direct current at a lower voltage than that supplied to the more remote main cathode. The impurities are deposited on the subsidiary cathode with only a small percentage of the metal, which is mainly deposited on the cathode proper.

E. S. KREIS.

Treatment of minerals. W. O. BORCHERDT, Assr. to NEW JERSEY ZINC CO. (U.S.P. 1,585,756, 25.5.26. Appl., 11.12.22).—The mineral pulp is first subjected to a froth-flotation process in which a substantial amount of the colloidal constituent is removed, and is then treated by a non-flotation method of separation. C. A. KING.

Flotation of ores. H. W. MORSE, Assr. to COMP. DU BOLES (U.S.P. 1,587,789, 8.6.26. Appl., 29.7.25).—A flotation agent consisting of a mixture of an alkali xanthate and an alkali chloride is used.

C. A. KING.

Aluminium alloy and method of manufacture. H. K. RICHARDSON, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,585,786, 25.5.26. Appl., 5.11.23).—Ductile alloys of aluminium and zirconium are made by electrolysing a mixture of unpurified bauxite and zirconium oxide, the quantity of the latter being insufficient to give more than 10% Zr in the alloy. C. A. KING.

Process of making acid-proof alloys. J. P. KEGG (U.S.P. 1,586,368, 25.5.26. Appl., 28.2.22).—Copper and lead are melted together, the latter being present in such quantity as to segregate. An alkali metal is added to the melt to hold the lead in suspension. C. A. KING.

Alloy. C. W. HEPPENSTALL, Assr. to HEPPENSTALL FORGE & KNIFE CO. (U.S.P. 1,587,231, 1.6.26. Appl., 7.3.25).—The alloy contains C 1·5—2·25%, Cr 3·5—6%, W 0·5—2%, the remainder being the usual components of commercial steel.

C. A. KING.

Combining molten metal with a gas. H. A. POPPENHUSEN (U.S.P. 1,586,328, 25.5.26. Appl., 26.3.24).—The gas is blown across the open end of a pipe communicating with a supply of molten metal, whereby the metal is drawn up into the pipe by the action of the blast and just sufficient metal is carried forward from the open end of the pipe to react with the blast. The blast is preheated by leading the supply pipes through the main mass of molten metal. E. S. KREIS.

Refining metals. S. WESTBERG (U.S.P. 1,586,543, 1.6.26. Appl., 21.10.24).—Impurities in ferrous material are removed by heating the material in a reducing atmosphere at normal pressure, in the presence of an alkaline-earth compound, to a welding temperature but below the melting point.

H. ROYAL-DAWSON.

Method of introducing additional agents into metallurgical baths. O. NIELSEN (U.S.P. 1,587,600, 8.6.26. Appl., 4.12.24. Conv., 4.12.23).—Inert gas is introduced into a metallurgical bath with the object of removing air from the pipe line to the bath, and the refining agent is then introduced through the same pipe line, in gaseous, liquid, or solid form. C. A. KING.

Purification of zinc solutions. T. P. CAMPBELL (U.S.P. 1,587,695, 8.6.26. Appl., 8.9.25).—Impure

acid or alkaline zinc solutions are passed upwards through a narrow pipe in the middle of a closed cylindrical vessel, overflow from the top into a compartment made by standing a vessel in the shape of a truncated cone on a screen disposed just above the bottom of the cylinder, and then pass upwards again between the walls of the cylinder and the cone. All the three compartments are packed with zinc shot or scrap, and by this arrangement the rate of flow of the solution is gradually diminished in its passage through the apparatus.

A. R. Powell.

Recovery of zinc and lead from zinc–lead ores. Soc. Gén. Métallurgique de Hoboken (F.P. 602,385, 24.11.24).—Zinc–lead ores are heated in rotating cylindrical furnaces to volatilise lead and zinc oxides, which are subsequently separated by leaching with ammonium carbonate solution. If the ore contains sulphur, sufficient copper material is added to retain the whole of the sulphur in the speiss. If both copper and sulphur are present the proportion of each is so regulated, either by addition of more copper or sulphur, or by roasting, that the whole of the copper and sulphur remain in the residue in the furnace.

A. R. Powell.

Recovery of nickel from ores. J. J. Hissink (F.P. 595,275, 18.3.25. Conv., 31.3.24).—Finely-divided nickel ores are extracted with gas liquor in the presence of a current of air. When the ore has previously been heated under reducing conditions, it must be allowed to cool in the presence of air so that it is re-oxidised and the nickel thus rendered soluble in the gas liquor.

L. A. Coles.

Electroplating with cadmium. Udylite Process Co., Assees. of M. E. Louth and A. W. Young (E.P. 235,159, 30.4.25. Conv., 5.6.24).—See U.S.P. 1,537,047 ; B., 1925, 554. A concentration of 2·5—10 oz. of free sodium cyanide per gall. of plating solution is specified.

Brass alloy. F. Heusler, Assr. to Isabellenhütte Ges.m.b.H. (U.S.P. 1,590,092, 22.6.26. Appl., 29.4.21. Conv., 2.2.17).—See G.P. 303,864 ; B., 1921, 265 a. The proportions specified are Cu 37, Si 13, Fe 20, Mn 30%.

Iron casting. J. E. Fletcher and H. J. Young (U.S.P. 1,589,062, 15.6.26. Appl., 18.3.26. Conv., 1.10.24).—See E.P. 245,196 ; B., 1926, 195.

Desulphurising iron. G. S. Evans, Assr. to Mathieson Alkali Works (U.S.P. 1,590,730, 29.6.26. Appl., 23.4.24).—See E.P. 232,963 ; B., 1925, 962.

Manufacture of metal, more especially magnesium, from the corresponding chloride. C. Arnold. From Dow Chemical Co. (E.P. 254,050, 7.5.25).—See U.S.P. 1,567,318 ; B., 1926, 196.

Silver alloy. F. Heusler, Assr. to Isabellenhütte Ges.m.b.H. (U.S.P. 1,590,091, 22.6.26. Appl., 29.8.21. Conv., 13.9.20).—See E.P. 169,144 ; B., 1922, 298 a.

Condensing apparatus in connexion with an electric furnace for the production of volatile metals. F. Juretzka (U.S.P. 1,590,521, 29.6.26. Appl., 20.4.23. Conv., 22.4.22).—See G.P. 385,893 ; B., 1924, 301.

Apparatus [furnaces] for use in the cementation of iron or iron alloys. W. H. Fisher and P. Chambers (E.P. 253,641, 30.3.25).

Increasing the efficiency of Cowper stoves in existing blast-furnace plants. Hauts Fourneaux & Aciéries de Differdange-St.-Ingbert-Rumelange Soc. Anon., and P. Ries (E.P. 249,063, 14.5.25. Conv., 16.3.25. Addn. to 245,058).

Froth-flotation apparatus. Minerals Separation, Ltd. From Minerals Separation North American Corp. (E.P. 253,618, 20.3.25).

Electromagnetic separators for the separation or concentration of minerals. M. Koizumi (E.P. 254,030, 3.4.25).

Electro-deposition of metals on wire or narrow strip. J. A. Parker (E.P. 254,067, 23.5.25).

Pyrometer sheath (E.P. 251,750).—See I.

Electric furnaces (E.P. 239,510).—See XI.

XI.—ELECTROTECHNICS.

Use of electrodes of V2A-steel in electroanalysis. A. Schleicher and L. Toussaint (Z. angew. Chem., 1926, 39, 822—824).—An investigation of the stability of an electrode of V2A-steel towards various acid, alkaline, and saline media, and of the conditions under which the technically important metals may be quantitatively deposited in a weighable form from aqueous solutions containing their salts when the customary platinum cathode is replaced by a gauze of V2A-steel. Since the steel is attacked by hydrochloric and sulphuric acids, determinations in presence of these acids are not possible. The electrode may, however, be used in the presence of nitric acid, a mixture of nitric and sulphuric acids, acetic, oxalic, and tartaric acids, salts of organic acids, cyanides, ammonia, or free alkali. Using such a cathode, copper, bismuth, nickel, iron, antimony, and tin may be determined quantitatively under the usual conditions for the quantitative electrodeposition of these metals on platinum. Silver is deposited from solutions containing nitric acid in the form of relatively large, non-adherent crystals. Excellent results are, however, obtained when the deposition is from alkaline cyanide solutions. Zinc is deposited quantitatively in a compact form from acetic acid solutions but not from alkaline solutions. Cadmium is deposited quantitatively from acetic acid solutions, but has a tendency to form loose crystals, and consequently an electrode with a large surface is necessary. The electrode may not be used for the determination of mercury or in any process involving heating of the

deposited metal or metal oxide, *e.g.*, anodic deposition of lead, manganese, etc. Provided that it is possible to obtain a sufficiently large surface, V2A-steel is an excellent substitute for platinum. The electrode surface is prepared by washing with nitric acid, water, and, finally, methyl alcohol. J. S. CARTER.

Electric furnaces for temperatures up to 3300°. LÖWENSTEIN.—See A., July, 706.

Indurating concrete with sulphur. KOBBÉ.—See IX.

Electric properties of condensation products of phenols and aldehydes.—SHONO.—See XIII.

Electrodeposition of rubber. ELLIOTT.—See XIV.

Electrolytic preparation of glycollic acid. TOJA and CEVA.—See XX.

PATENTS.

Electric discharge tubes with rarefied atmospheres. J. B. J. M. ABADIE and N. M. COURTINES (E.P. 230,467, 4.3.25. Conv., 4.3.24).—The pressure inside an electric discharge tube with a rarefied atmosphere functioning with high frequency or high tension may be maintained constant by providing a substance adapted to emit a vapour or gas corresponding to the atmosphere of the tube. The required vapour tension may be reached at ordinary temperatures or, if at higher temperatures, the heating is effected by cathodic bombardment, the substance being placed in a hollow electrode provided for the purpose. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 225,890, 210,728, 5557 of 1915, 12,256 and 1218 of 1913, and 29,839 of 1912.) M. E. NOTTAGE.

Electric discharge vessels, tubes, and the like. E. F. HUTH GES. FÜR FUNKENTELEGRAPHIE M.B.H. (E.P. 230,492, 9.3.25. Conv., 8.3.24).—An electron-emitting cathode, in an electric discharge vessel of the dull emitter type, if made of an alloy of molybdenum and thorium, is capable of emitting electrons without any heat treatment being employed for rendering it active. An electrode of molybdenum is mounted in the discharge vessel and heated therein for a short time before or after being coated with thorium nitrate, the electrode being produced without the use of a binding or reducing agent. The electron emission of this electrode takes place at comparatively low temperatures, *e.g.*, about 1200°, whereby its life is prolonged; also the heating current is smaller than that required for heating a tungsten or tungsten-thorium cathode of the same specific emissivity. M. E. NOTTAGE.

Making sag-resisting bodies [filaments] of tungsten. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of S. DUSHMAN and P. A. KOBER (E.P. 244,116, 4.12.25. Conv., 8.12.24).—Change of structure and shape of coiled tungsten filaments at the high temperatures attained in gas-filled incandescence lamps and similar devices may be prevented by heating the filaments to within the temperature range of rapid grain growth, while supported so that every part is free from stress great enough to cause deformation. M. E. NOTTAGE.

Leading-in wire for glass vessels [electric lamp bulbs etc.]. E. FRIEDERICH, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,588,179, 8.6.26. Appl., 30.8.21. Conv., 10.2.16).—The wire is composed of an alloy of iron and tungsten having a coefficient of expansion approximating to that of glass. C. O. HARVEY.

Electric furnaces. SIEMENS & HALSKE A.-G. (E.P. 239,510, 28.8.25. Conv., 2.9.24).—In an electric induction furnace for smelting metals the heating body, which forms part of the secondary winding of a transformer, is in the form of an annular hollow pipe of rectangular cross-section, and consists, on the inner side, of a fireproof material, *e.g.*, silit, and on the outer side of a metal. The silit walls are made up of separate plates which may be brought close together and insulated from one another. This hollow pipe completely encloses the primary winding which consists of copper pipes, through which water circulates constantly, wound on an annular iron core made up of plates; by this arrangement no appreciable currents are induced in the material to be treated. The primary winding is divided into sections which can be connected in series or in parallel, thus facilitating regulation of the furnace. The metal to be smelted is placed in the centre of the annulus in a crucible; or, alternatively, the inner wall of the annulus formed by the heating body may form part of the receptacle. M. E. NOTTAGE.

Electric furnaces. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of C. L. IPSEN (E.P. 241,897, 19.10.25. Conv., 23.10.24).—In an electric resistor furnace with a heat-refractory wall forming the heating chamber, the highest possible safe operating temperature, without injury to the heating resistor, may be obtained by mounting the resistor directly in the heating chamber. The resistor, *e.g.*, a nickel-chromium alloy, is bent or cast into a series of convolutions, the upper bends of which are secured, by means of metallic rings, to a bar made of insulating material fixed near the top of the furnace. The lower bends are secured in the same way, but the bar is not fixed to the furnace wall so that the resistor is free to expand or contract; also, the weight of this bar keeps the resistor straight. M. E. NOTTAGE.

Flame-proof insulating material. WESTERN ELECTRIC CO., LTD. From WESTERN ELECTRIC CO., INC. (E.P. 252,856, 1.5.25).—The material is made by intimately mixing together 25 pts. of chlorinated naphthalene (preferably tetrachloronaphthalene), 20 pts. of rubber, 33 pts. of powdered silica, 2 pts. of sulphur or other vulcanising agent, and 10 pts. of litharge or other substance capable of accelerating vulcanisation. The resulting mixture is sufficiently plastic to be moulded or to be extruded upon an electrical conductor in strand form. After being

moulded or extruded it is subjected to heat and pressure to vulcanise the rubber. Chlorinated naphthalene will not decompose or burn unless subjected to an intense heat, and silica has a high thermal conductivity. M. E. Nottage.

Electrolytic tank. L. Arnalot (U.S.P. 1,586,435, 25.5.26. Appl., 5.12.23).—An electrolytic tank is connected with a refrigerator and a receiving tank, liquid being passed from the receiving tank, through the refrigerator, and thence to the electrolytic tank, and back from the electrolytic tank to the receiving tank by means of the gas pressure generated within the electrolytic tank. M. E. Nottage.

Production of gas by electrolysis. R. L. Walsh (U.S.P. 1,588,214, 8.6.26. Appl,. 29.6.21).—The current is supplied through a number of electrodes. Means are provided for depolarising the electrodes simultaneously by washing, and for regulating the current density. C. O. Harvey.

Rendering a porous powder [used in an electric cell] impermeable to liquids. R. Oppenheim, Assr. to Soc. Anon. le Carbone (U.S.P. 1,588,607, 15.6.26. Appl., 10.4.24. Conv., 27.12.23). —See E.P. 226,769; B., 1925, 555.

Galvanic battery. R. Oppenheim, Assr. to Soc. Anon. le Carbone (U.S.P. 1,588,608, 15.6.26. Appl., 20.5.24. Conv., 10.4.24).—See E.P. 230,307; B., 1925, 364.

Portable ozonisers. E. L. R. Cailliet and M. L. J. Bourdais (E.P. 246,841, 26.1.26. Conv., 29.1.25).

Electrical apparatus for generating ozone. J. McBlain (U.S.P. 1,588,976, 15.6.26. Appl., 12.4.24. Conv., 24.4.23).—See E.P. 219,387; B., 1924, 795.

Metallic compositions. [Lead amalgams for storage-battery plates.] A. Miller (E.P. 254,209, 12.1.26).—See U.S.P. 1,570,763; B., 1926, 496.

Electrolytic production of hydrogen peroxide (F.P. 603,043).—See VII.

Electrolytic deposits of chromium (E.P. 243,667).—See X.

Aluminium alloy (U.S.P. 1,585,786).—See X.

Electrolytic production of metals (U.S.P. 1,567,791).—See X.

Electrometric determination of acidity or alkalinity of soils, waters, etc. (G.P. 413,043).—See XVI.

XII.—FATS; OILS; WAXES.

Oil extraction in theory and practice. L. C. Whiton (Ind. Eng. Chem., 1926, **18**, 605—606).—An examination of the practical working of extraction plants shows that many features theoretically excellent are undesirable. Simplicity of construction with absence of movable parts, units of small size, and individual extractors as opposed to a series arrangements are advocated. D. G. Hewer.

Olive oil. I. F. Traetta-Mosoa (Annali Chim. Appl., 1926, **16**, 169—177).—The best olive oil, as regards aroma, fluidity, and stability, is obtained from slightly immature olives, which give also a good yield. If the fruit is too unripe, the oil is bitter and the yield poor; fully ripe olives furnish good oil, but fruit left too long on the trees produces a dense, discoloured oil which readily turns rancid. Between gathering and pressing the olives should be kept spread in thin layers in a cool place, since if heaped or trenched they become heated to 35—50° and undergo such fermentative changes that the resulting oil is of bad odour and persistently turbid. A sample of oil from worm-eaten and fermented olives contained lipolytic and proteolytic enzymes and yielded three varieties of *Saccharomyces*, the morphological characters of which are described. As regards analytical results, the most important differences between oil from sound and that from altered olives lie in the total acidity and in the volatile and soluble acid value. For good oils the acid value ranges from 1·02 to 1·22, whereas with the altered oils it varies between 3·04 and 5·58; the volatile and soluble acid value is 1·10—1·15 and as high as 3·6 in the two cases. T. H. Pope.

Sensitive reaction for olive oil extracted with carbon disulphide. P. Saccardi (Giorn. Chim. Ind. Appl., 1926, **8**, 11).—In this reaction use is made of a filtered solution of 5 g. of lead soap in 100 c.c. of benzene, which keeps well, and of a solution of 30 g. of potassium hydroxide in 100 g. of 95% alcohol, this being filtered and stored in glass-stoppered bottles. If 1 c.c. of an oil which has been obtained by extraction with carbon disulphide is heated to boiling with 1 c.c. of the alcoholic potassium hydroxide solution and 1 c.c. of the lead soap solution, intense blackening, followed by precipitation of lead sulphide, takes place. This method detects about 20 g. of the so-called sulphur oil per litre of olive oil, or, if the oil is shaken with 25% of 95% alcohol and distilled, and the distillate tested, about 10 c.c. of the sulphur oil per litre. This test is not invalidated if the oil is previously either heated at 130° or treated with superheated steam for 2 hrs. Traces of carbon disulphide vapour can also be detected in this way. T. H. Pope.

Oil extracted from the head of a dolphin. H. Marcelet (Compt. rend., 1926, **182**, 1416—1417).—The oils obtained from (1) the maxillary glands, (2) the nose, and (3) the head of a dolphin (*Delphinus delphis*, Lin.), although generally similar in type, exhibited well-marked differences, *e.g.*, d^{15} (1) 0·9206, (2) 0·9308, (3) 0·9330; n_D^{17} (1) 1·4548, (2) 1·4640, (3) 1·4790; saponif. value (1) 267, (2) 259, (3) 212; iodine value (Wijs) (1) 17, (2) 56, (3) 133; soluble volatile acids expressed as Reichert value (1) 145·3, (2) 111·3, (3) 39·1; unsaponif. matter (1) 16·30%, (2) 6·07%, (3) 1·77%; solid

fatty acids (1) 30·82%, (2) 19·28%, (3) 10·08%; liquid fatty acids (1) 18·70%, (2) 43·17%, (3) 74·04%.
H. J. EVANS.

Detection of α- in presence of β-palmitodistearin. [Detection of tallow etc. in lard.] F. J. F. MUSCHTER and G. VISSER (Chem. Weekblad, 1926, **23**, 250—252).—The presence of tallow and other fats in lard can be detected with more certainty by microscopical examination than by determination of the Boemer value (cf. B., 1926, 637). Staining with Romanowsky–Giemsa solution (azur-eosin in alcohol) greatly facilitates the detection of the isomeric palmitodistearins. Many photomicrographs are reproduced.
S. I. LEVY.

Determination of milk fat in mixtures of fats. J. GROSSFELD (Z. Unters. Lebensm., 1926, **51**, 203—213).—The determination of the butyric acid number, from which the proportion of milk fat in a mixture can be calculated (cf. B., 1926, 447), is influenced by the solubility of the butyric acid in the non-aqueous phase, especially in liquid or molten insoluble fatty acids. Hence in the case of oils containing a considerable proportion of liquid fatty acids, it is preferable to remove these as magnesium soaps by means of magnesium sulphate and then add a corresponding quantity of fatty acids (in the form of soap) having a butyric acid number 0, before determining the butyric acid number.
B. W. CLARKE.

Detection of linseed oil in soya bean oil. J. F. CARRIÈRE (Chem. Weekblad, 1926, **23**, 274—279).—Whilst the iodine value or the hexabromide number alone may not be sufficient, a consideration of the two together will serve for the detection. The hexabromide number is best determined on the oil itself, not on the free fatty acids, and a suitable procedure is described. For linseed oils the relation between the iodine value, I, and the hexabromide number, H, is given from many determinations as $I—126·29=1·574H$, whilst for soya bean oil the corresponding relation is $I—126·19>12H$. Formulæ are deduced from these relations by means of which the linseed oil content of a mixture may be determined from the values I and H within moderate limits, provided marine animal oils be not present.
S. I. LEVY.

Chemistry of drying oils. III. G. W. ELLIS (J.S.C.I., 1926, **45**, 193—199 T; cf. B., 1925, 768, 930).—A further study of linoxyn from linseed oil and of the oxy-acids obtained from the unsaturated acids of this oil is described. On both products a number of determinations of the hydroxyl groups and of the ketone groups were carried out by means of Grignard's reagent and phenylhydrazine respectively. The number of groups obtained were calculated to the formula $C_{57}H_{96}O_{20}$ representing, or closely approximating to, that previously assigned to linoxyn, whilst in the case of the oxy-acids they were calculated to a mixture representing 1 mol. of oxylinolenic acid to 2 mols. of oxylinoleic acid. The results showed that linoxyn contained about 7 hydroxyl groups and the mixed oxy-acids about 10 hydroxyl groups. Since the reagent reacts with acidic hydroxyl groups the two series of results were in agreement. The number of ketone groups present in linoxyn and in the oxy-acids amounted to 6 or 7. Hence the 14 atoms of oxygen introduced during the autoxidation process were, for the most part, accounted for. The explanation offered for the presence of these groups is that during the autoxidation process an atom of oxygen is introduced between each ethylene carbon and its accompanying hydrogen atom, with formation of an enol grouping which may give rise to a ketol:

·CH : CH· → ·C(OH) : C(OH)· → ·CO·CH(OH)·.

The enol forms explain the susceptibility of linoxyn and of the oxy-acids to hydrolysis with the production of aldehydes and acids and the resinous products of the former. Other properties of linoxyn such as its behaviour towards solvents and its ready solubility in the molten phenols are given in evidence for the presence of the atomic grouping C·OH. Possible formulæ for oxylinoleic and oxylinolenic acids are suggested.

Hydrogenation of cottonseed oil with platinum. Heterogeneous catalysis. III. A. S. RICHARDSON and A. O. SNODDY (Ind. Eng. Chem., 1926, **18**, 570—571).—Hydrogenation of refined cottonseed oil between 40° and 240° with platinum catalyst is not so selective as with nickel catalyst (B., 1924, 564; 1925, 137), although the proportion of stearic acid formed is substantially less than would be expected if hydrogenation of linoleic to oleic and of oleic to stearic acid proceeded continuously at rates proportional to the concentration of the reacting acids. Partial hydrogenation with platinum to a given point would produce a more readily oxidisable product than with nickel. Rise of temperature was found to favour selective hydrogenation.
D. G. HEWER.

Detection of traces of nickel in hardened fats. M. WAGENAAR (Pharm. Weekblad, 1926, **63**, 570—575).—The fat is melted in a small porcelain basin, and a strip of (analytical) filter paper, rolled into a cylinder round a thin platinum wire, is inserted vertically to serve as a wick, at the top of which the fat is burned. When no fat is left, the paper is burned to ash in a silica or platinum crucible, and the residue damped with acid and tested for nickel with dimethylglyoxime. 0·03 mg. in 30 g. of fat can be detected with certainty.
S. L. LEVY.

Determination of concentration of liquid soaps by the immersion refractometer. L. F. HOYT and A. VERWIEBE (Ind. Eng. Chem., 1926, **18**, 581—582).—The Zeiss immersion refractometer is particularly adapted for the routine control analysis of liquid soaps of known fat composition. In the case of aqueous solutions of potassium soaps made from various thoroughly saponified fats the refractive index was found to be directly proportional to the content of total solids, which may be rapidly calculated to an accuracy of 0·05%. The refractometer can be

read to 0·1°, which corresponds to about 0·025% of soap solids, and accuracy is obtained to the limit of the readings (20—22% of total solids). The slope of the curve of concentration versus the refractive index of the liquid potassium soaps of various oils (cottonseed, maize, olive, linseed) is proportional to the refractive indices of the oils whether measured at 20° or 40°, but the corresponding results with soaps prepared from fatty acids are not so consistent. The concentration of the total solids may be calculated from the equation $y=ax+k$, where y is the refractive index at 20°, x the % of soap solids, k is 1·33299 (the refractive index of distilled water at 20° on the Zeiss instrument), and a is the numerical value of the slope, ranging from $1{\cdot}425\times10^{-3}$ for coconut oil to $1{\cdot}561\times10^{-3}$ for linseed oil. D. G. HEWER.

See also A., July, 672, **Relative concentrations of various electrolytes required to salt out soap solutions** (MCBAIN and PITTER). 712, **X-Ray identification of higher fatty acids** (MORGAN and HOLMES); **Stereoisomerism of ethylenic acids. Hydrogenation of stearolic and behenolic acids** (GONZALEZ).

Measuring viscosity at higher temperatures. MERCER.—See II.

Fatty acids associated with maize starch. TAYLOR and LEHRMAN.—See XVII.

PATENT.

Centrifugal liquid purifiers (E.P. 252,421).—See I.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Analysis of Prussian blues. T. H. BOWLES and J. F. HIRST (J. Oil and Colour Chem. Assoc., 1926, **9**, 153—156).—A scheme for the complete analysis of Prussian blue is recommended. It comprises determinations of ferric iron (cation), ferrocyanide radical, alkalis, total iron, hygroscopic and combined water, and water-soluble impurities. The results of analyses of 5 English, 1 German, and 2 American samples are given, and the influence of the alkalis present is discussed, the combined water content of potash and ammonia blues being considerably lower than that of the soda blues (of inferior colouring power). The determination of the iron–cyanogen complex is a more satisfactory criterion of the value of a Prussian blue than is the ash. Support is given to the view that the pigment is mainly ferric ferrocyanide rather than ferrous ferricyanide. S. S. WOOLF.

Titanium white. C. P. VAN HOEK (Farben-Ztg., 1926, **31**, 2296—2298).—A summary of the hitherto published information on the chemical and physical properties of the mixtures of titanium dioxide and barium sulphate sold as titanium white. A comprehensive bibliography is given. S. S. WOOLF.

Alteration of paint films on heating. H. WOLFF and G. ZEIDLER (Korrosion u. Metallschutz, 1926, 95; Farben-Ztg., 1926, **31**, 2300).—Tensile strength and extension determinations were carried out on detached paint films that were heated at 80° for various lengths of time. In all cases the tensile strength per gram increased and the percentage extension decreased on heating. In the case of films of white lead and lithopone paints the tensile strength first rose rapidly, then much more slowly, until the film became brittle and crumbled. Zinc white and red oxide of iron films, on the other hand, showed a steady rise to a maximum, after which a loosening of the structure took place, the tensile strength falling with eventual disintegration of the film. These phenomena are attributed to colloid-chemical changes in the film rather than to the chemical nature of the pigments used. S. S. WOOLF.

Protective paint coatings. J. N. TERVET (J. Oil and Colour Chem. Assoc., 1926, **9**, 157).—The exposure panel previously described by the author—a steel panel coated with metallic lead primer, followed by green paint (cf. J.S.C.I., 1925, 28 T)—has been re-examined after a further exposure of 18 months, making 3 years 3 months in all. Complete protection is still afforded by the coating as metallic lead is seen in close contact with the steel when the film is broken. While rust is apparent where the steel has been exposed in previous examinations, there has been no creeping of the rust under the film of primer. S. S. WOOLF.

Effect of various carbon pigments upon the rate of oxidation of linseed oil. F. H. RHODES and H. E. GOLDSMITH (Ind. Eng. Chem., 1926, **18**, 566—570).—The marked inhibition of the oxidation of raw linseed oil which occurs on addition of lampblack or carbon black is due to the continuous adsorption of the intermediate oxidation product which is the true catalyst in the drying reaction. With paints prepared with cobalt drier the inhibiting effect of carbon is more marked on keeping, as the pigment adsorbs the cobalt drier; with lead drier oxidation is nearly entirely inhibited during the first hours of exposure, as the small amount of autocatalyst originally present in the oil or produced during the initial period appears to be adsorbed, as well as a large part of the lead drier; with manganese drier the rate of oxidation is scarcely affected by carbon pigments. D. G. HEWER.

Capillary-analysis and its application to the examination of resins. E. STOCK (Farben-Ztg., 1926, **31**, 1903—1904, 1959—1960, 2133—2134, 2187—2188, 2240—2243).—On drying filter-paper strips that have been partially immersed in resin solutions for periods up to 24 hours, a series of "adsorption-pictures" result, showing strata of different colour, extent, intensity, opacity, etc. When this test is carried out under specified conditions in respect of dimensions and quality of filter-paper, strength of solution, depth and time of immersion, size of vessel, temperature, relative humidity, etc., the critical examination of the zones in the adsorption-pictures obtained furnishes information as to the identity and purity of the resin, the existence of

admixed resins, whether advantageous or adulterant, etc. A series of 48 reproductions (in black and white) of the results of tests on resins and resin mixtures is given, with full descriptions in the text.

S. S. Woolf.

Condensation products of phenols and aldehydes. III. Electrical properties. T. Shono (J. Soc. Chem. Ind. Japan, 1926, 29, 121—126).—The surface colour of the phenol–formaldehyde condensation product prepared by using ammonia and potassium hydroxide as the condensing agents under ordinary pressure is darkened by exposure to an oxidising atmosphere, especially on heating. The mechanism of the condensation seems to be different according to the nature of the condensing agents used. When the condensation product is heated in an oxidising atmosphere, the weight and volume of the material diminish, and the interior colour fades; the capacity for acquiring an electric charge increases nearly to the same degree as the deepening of the surface colour, and the dielectric strength is increased. (Cf. B., 1926, 595.)

K. Kashima.

Chemistry of drying oils. Ellis.—See XII.

Patents.

Titanium pigments. J. Blumenfeld and M. Mayer (E.P. 252,262, 28.11.24).—Titanium oxide is suspended in a solution containing one of the rare earths of the titanium–thorium group (*e.g.*, the solution of titanium sulphate obtained by treating ilmenite with sulphuric acid). On heating, oxides or hydroxides are precipitated on to and seal the pores of the titanium oxide base. The titanium pigment so produced does not "chalk" on weathering. An alternative method is to mix the titanium oxide with an aqueous colloidal solution of oxide of zirconium, thorium, or titanium, and then evaporate the water. After neutralising any acids present the mixed product is calcined at red heat.

S. S. Woolf.

Fillers for [lake] pigment colours, rubber, etc. W. Eberlein, and Colloisil Colour Co. (E.P. 252,416, 16.1.25).—A colloidal suspension of a colour-fixing earth, such as fuller's earth, green earth, white earth, bentonite, or any natural or artificial silicate that absorbs and fixes basic dyes, is mixed with an organic substance (excluding basic dyes), *e.g.*, amino- or hydroxy-compounds, oils, fats, waxes. After drying a non-oily, fine, soft powder is obtained which still retains the property of fixing basic dyes and may be used as a filler for lake pigments, rubber, etc.

S. S. Woolf.

Treatment of zinc white or similar metallic oxides, lithopone, and white lead for use in the preparation of paint. A. van Lerberghe (E.P. 252,756, 2.1.25).—The pigment in the form of an aqueous paste is ground with a drying oil containing free fatty acid, whereby the excess of water separates and saponification occurs with formation of metal soaps. For example, 220 lb. of zinc oxide are ground in 155 lb. of water for ½ hr., 30 lb. of drying oil containing 6—7% of admixed linoleic acid are added, and grinding is continued for a further ¼ hr. The resulting zinc linoleate paste is crushed to a smooth non-setting paste capable of being readily diluted for use in paint.

S. S. Woolf.

Self-disintegrating metallic compounds for use in anti-fouling paints. W. P. Heskett and H. B. Molesworth (E.P. 252,766, 26.2.25).—One or more of the metals specified in E.P. 158,740 (B., 1921, 224 A) is fused with one or more of the compounds of these metals, to give a self-disintegrating material which may be incorporated in a suitable medium to form an anti-fouling paint, since it is decomposed by water to yield a poisonous gas. A typical mixture is iron oxide, iron sulphide, and aluminium, which will evolve hydrogen sulphide. On introducing antimony or arsenic or arsenical pyrites into the mixture, stibine or arsine is produced together with the hydrogen sulphide. The material may be pulverised mechanically and converted into paint by means of one of the carriers described in E.P. 226,286 (B., 1925, 107).

S. S. Woolf.

Composite [green] pigment. H. Dourif (U.S.P. 1,587,704, 8.6.26. Appl., 1.4.24).—Solutions of a chromate and of a metal salt capable of reacting therewith to form an insoluble chromate are added to a non-settling suspension of ultramarine blue, whereupon a composite pigment is precipitated.

S. S. Woolf.

[Rubber] paint. G. A. Shine (U.S.P. 1,588,150, 8.6.26. Appl., 3.6.25).—Devulcanised old rubber is fused with fossil resin, with or without the addition of hydrated lime, and the whole dissolved in a rubber solvent. 10% by weight of zinc sulphate may be added to the thinned mixture.

S. S. Woolf.

Polymerising vinyl esters. Consortium für Elektrochem. Ind., Assees. of W. O. Hermann and E. Baum (U.S.P. 1,586,803, 1.6.26. Appl., 8.8.25. Conv., 13.8.24. Cf. G.P. 281,687–8; B., 1915, 623).—Vinyl esters are readily polymerised to clear hard resins by heating under pressure with water and a catalyst consisting of an organic or inorganic peroxide, a per-salt, a per-acid, or a mixture of such compounds. The action of hydrogen peroxide and of organic peroxides is increased by addition of alkaline substances, and the properties of the product may be modified by performing the reaction in presence of an organic solvent such as glycol, glycerol, paraldehyde, or ethylidene diacetate. Polymerisation may also be effected by heating the vinyl ester with water alone under pressure or by treating it with water in presence of radiant energy.

T. S. Wheeler.

Process of treating resins. C. F. Willard (U.S.P. 1,587,622, 8.6.26. Appl., 21.5.24).—Resin is heated with glycerol, rubber, and a suitable catalyst until the resin has melted and combined with the rubber and glycerol to form a resin gum ester.

D. F. Twiss.

[Paracoumarone] varnish. W. W. King, Assr. to Barrett Co. (U.S.P. 1,587,333, 1.6.26. Appl., 9.2.21).—A drying oil is heated to about 240°, paracoumarone resin is added, and the temperature is raised to a higher degree and maintained thereat until the mixture will set when cooled; further paracoumarone is then added. S. S. Woolf.

Manufacture of titanic and zinc compounds [pigments]. P. Pipereaut and A. Helbronner (U.S.P. 1,590,697, 29.6.26. Appl., 21.11.23. Conv., 25.11.22).—See E.P. 207,555; B., 1925, 107.

Sulphur phenol resins. Soc. of Chem. Ind. in Basle, Assees. of A. Blumfeldt (U.S.P. 1,588,439, 15.6.26. Appl., 31.7.23. Conv., 1.9.22).—See E.P. 203,310; B., 1924, 566.

[Control of stirrer in] apparatus employed in synthetic resin condensation processes. Brit. Thomson-Houston Co., Ltd., H. W. H. Warren, R. Newbound, and L. M. T. Bell (E.P. 253,614, 20.3.25).

Apparatus for testing the viscosity of fusible materials [shellac]. Metropolitan-Vickers Electrical Co., Ltd. From Westinghouse Electric and Manuf. Co. (E.P. 254,239, 26.3.26).

Colour lakes (U.S.P. 1,587,435).—See IV.

Obtaining hydrogen peroxide and *blanc fixe* (E.P. 252,768).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Application of disinfectants used in the cultivation of rubber. A. Steinmann and J. J. B. Deuss (Comm. Proefstat. Rubber, Buitenzorg, 1926, [3], 159—197; Arch. Rubbercultuur, 1926, [5]).—Protection of rubber trees against infectious diseases is commonly effected by the application of certain forms of tar or preparations of tar. Various types of these disinfectants are described together with methods for their examination in the laboratory and on the tree. D. F. Twiss.

Specific gravity of *Hevea* latex. V. O. de Vries (Comm. Centr. Rubber Stat. Buitenzorg, 1926, [53], 1—23; Arch. Rubbercultuur, 1926, [1]).—It is probable that all earlier statements as to *Hevea* latex with a specific gravity exceeding that of water are in error. Undiluted latex, the rubber content of which, because of heavy tapping or other physiological factors, decreases below 17%, may actually show a specific gravity exceeding 1. For example, latex from one tree contained 14% of rubber and less, and its specific gravity reached 1·004 and higher. With small deviations the specific gravity accorded with that expected for a mixture of serum and rubber with specific gravity 1·020 and 0·914 respectively. D. F. Twiss.

Preservation of [rubber] latex with ammonia combined with other means. O. de Vries (Comm. Proefstat. Rubber, Buitenzorg, 1926, [2], 149—157; Arch. Rubbercultuur, 1926, [4]).—It is not possible to effect an economy by using a reduced proportion of ammonia to neutralise natural acidity together with some additional means of checking the growth of micro-organisms. Examination was made of the effect of heat, formalin, and potassium cyanide respectively on latex containing less ammonia than customary. D. F. Twiss.

Native rubber. W. Spoon (Comm. Centr. Rubber Stat. Buitenzorg, 1926, [54], 29—106; Arch. Rubbercultuur, 1926, [2]; cf. B., 1925, 891).—A summarised report on the examination of 253 samples of "native" rubber from various parts of the Outer Provinces of the Dutch East Indies. D. F. Twiss.

Molecular magnitude of caoutchouc and gutta-percha. E. Ott (Naturwiss., 1926, **14**, 320; Chem. Zentr., 1926, I, 3400).—The extensibility of rubber is not necessarily evidence of the simultaneous presence of an amorphous and a crystalline phase. By *X*-ray investigation the maximal formula for caoutchouc is found to be $(C_5H_8)_8$. Gutta-percha is also found to be crystalline and to have a maximal formula $(C_5H_8)_{12}$. D. F. Twiss.

Constitution of high polymerides. E. Geiger (Diss., Zurich, 1926; Gummi-Ztg., 1926, **40**, 2143—2144).—In the formation of the colloidal condensation products of dimethylketen with arylcarbimides, the primary complexes, which are probably of open-chain structure, combine to form groups containing 5—9 of the initial complexes. Rubber when heated in ether at 250° yield an amorphous poly*cyclo*-compound (*d* 0·992) containing one ethylene linkage to 5 isoprene nuclei; the product from gutta-percha is identical with this. In the pyrogenic decomposition of rubber (or gutta-percha) the more volatile products such as isoprene and dipentene arise from the decomposition of the caoutchouc molecules, whereas the less volatile products obtained at a somewhat higher temperature, result from the decomposition of poly*cyclo*caoutchouc (or poly*cyclo*gutta) which has been formed previously. On hydrogenation at 270° rubber gives a mixture of two saturated compounds derived from caoutchouc and polyhydrocaoutchouc respectively; gutta-percha and balata hydrocarbons appear to yield identical products. Similarly metastyrene is reducible to hydrometastyrene without resolution into styrene. These results indicate that chemical valencies must be involved in linking the primary complexes to produce the colloid. Caoutchouc tetrabromide reacts with triphenylphosphine forming amorphous products which are strongly ionised in solution and undergo double decomposition with an alkali picrate. The oxidation products of rubber and of hydrocaoutchouc also support the view that the colloid structure of these substances is dependent on chemical valency. The earlier observation (Klein and Stamberger, B., 1925, 462) that rubber, after being milled, gives solutions containing particles visible in the ultramicroscope, is incorrect. D. F. Twiss.

Electrodeposition of rubber. F. A. ELLIOTT (Chem. Met. Eng., 1926, 33, 358—359).—With electrodes 4 in. apart, voltage 110, and current density at anode 0·4 amp./sq. in., a bath mixture of rubber (from latex) 8·0 grms., sulphur 0·3, zinc oxide 1·5, whiting, 4·5, carbon black 0·3, paraffin wax 0·3, tetraethylthiuram disulphide 0·03, gum arabic 0·075, and ammonia 0·16 g. per 100 c.c., gave in one minute a deposit 0·03 in. thick, of practically the same composition as the total solids of the bath. When dried and vulcanised the product was at least as tough and strong as an ordinary product of similar composition. The possibilities of the Sheppard and Eberlin process (cf. B., 1925, 681; also U.S.P., 1,580,795; B., 1926, 639) are reviewed.

D. F. TWISS.

Reaction between caoutchouc and sulphur. G. S. WHITBY and R. S. JANE (Trans. Roy. Soc. Canada, 1926, [iii], 20, III, 121—131).—Experiments carried out at various temperatures, in various solvents, and in the presence and in the absence of accelerators, show that, however prolonged be the period of heating, and however great the excess of sulphur used, the reaction product, after being freed from uncombined sulphur by the usual method of exhaustive acetone extraction always contains about 32% S, corresponding with $(C_5H_8S)_n$. Accelerators have a much smaller effect on the rate of vulcanisation in solution than when used in a dry rubber–sulphur mixture. Removal of protein and resin from rubber reduces the rate of combination with sulphur but is without effect on the 32% limit. Prolonged extraction of the caoutchouc–sulphur reaction product with alcoholic alkali gradually removes sulphur, the rate of removal being different for products obtained in different solvents; products obtained in halogenated aromatic hydrocarbons are more resistant than those obtained in aromatic hydrocarbons. After exhaustive extraction with fairly concentrated alcoholic alkali the products still contain about 19% S. The caoutchouc–sulphur reaction product cannot bring about vulcanisation, Ostromislenski's theory (B., 1916, 370) being untenable.

J. S. CARTER.

Latex viscosimeter. R. DITMAR (Chem.-Ztg., 1926, 50, 497—498).—A viscosimeter for rapid measurements of the viscosity of rubber latex consists of a wide glass tube, graduated at 100 and 200 c.c., slightly expanded at the top to hold a ring of cloth (which serves to filter off any coagulated rubber), and tapering at the bottom to a narrower tube which is attached by means of a rubber tube, fitted with a pinchcock, to a tube drawn out to a fine jet, the diameter of which varies according to the type of latex under examination (1 mm. for natural 38% latex). The time of flow of the first or second 100 c.c. is compared with the corresponding time for water in the usual manner.

J. W. BAKER.

PATENTS.

Vulcanisation of rubber. BRIT. DYESTUFFS CORP., C. J. T. CRONSHAW, and W. J. S. NAUNTON (E.P. 253,197, 12.3.25).—The dixylylguanidines, particularly the three di-*m*-xylyl compounds, are more active accelerators of vulcanisation than the lower homologues hitherto used.

D. F. TWISS.

Removing sulphur from vulcanised rubber. L. L. ODOM (U.S.P. 1,587,408, 1.6.26. Appl., 7.1.25).—Material containing vulcanised rubber is distilled through a heated substance having a stronger affinity for sulphur than rubber.

D. F. TWISS.

Rubber-vulcanisation accelerator. W. SCOTT, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,586,121, 25.5.26. Appl., 9.9.25).—The compounds obtained by the condensation of 2 mols. of a secondary amine such as piperidine, diethylamine, tetrahydro-2-methylquinoline, or piperazine with 1 mol. of an aldehyde, *e.g.*, formaldehyde, acetaldehyde, acraldehyde, or aldol, may be further condensed with 1 mol. of carbon disulphide to yield products which are of value as accelerators for either hot or cold vulcanisation of rubber. The substance $CH_2(C_5H_{10}N)_2$ formed by the condensation of piperidine and formaldehyde forms an oil, b.p. 237°; it reacts with 1 mol. of carbon disulphide to yield a crystalline compound. Dibenzylamine and formaldehyde yield a substance, m.p. 99°, which condenses with carbon disulphide to give a compound, m.p. 64°. 2 mols. of diethylamine and 1 mol. of formaldehyde give bisdiethylaminomethane, which yields a liquid product on treatment with carbon disulphide.

T. S. WHEELER.

Fillers for rubber (E.P. 252,416).—See XIII.

Rubber paint (U.S.P. 1,588,150).—See XIII.

XV.—LEATHER; GLUE.

New and old theories of the bating process. R. H. MARRIOTT (J. Soc. Leather Trades Chem., 1926, 10, 132—142; cf. Wilson, B., 1921, 92 A).—Experiments have shown that skins can be limed until only traces of elastin remain and the skin can be fallen, but yet not properly bated, the grain not being silky nor the skin flaccid. Ammonia tends to give flaccidity, by causing the separation of fibres into fibrils. The mere production of minimum swelling and the separation of fibres into fibrils, however, does not suffice to produce flaccidity and it is probable that it is necessary to remove degraded collagen also. The porosity of a bated skin seems to be connected with the cleansing of the hair follicles rather than with the mere splitting of the fibres. The elastin theory of bating does not account for the supple nature of the tanned skin, and the latter is only partially dependent on the degree of plumpness of the fibres during the tanning process. Excellent leathers can be obtained which contain all their elastin. There appears to be a connexion between the flaccidity and resilience of the bated skin and the softness of the resulting leather. Ammonia does not, but trypsin does, digest elastin, produce a silky grain, give porosity, and a more flaccid skin and rapidly hydrolyses

degraded collagen. Experiments show that during the liming process the collagen of the fibres is altered so that they begin to stain similarly to gelatin. During the bating this property is destroyed. The degraded form of collagen does not dissolve entirely in warm water. The presence of a degraded form of collagen in the skin tends to stiffness in the leather. The hydrolysis of collagen to peptone takes place in at least four steps, α-collagen→β-collagen→gelatin→protcose→peptone. Pepsin can readily change α-collagen into β-collagen, but it cannot hydrolyse β-collagen so rapidly. Trypsin cannot hydrolyse α-collagen, but rapidly dissolves the β-form. β-Collagen is probably produced during the liming process and although it may be dissolved by the lime liquor, more is produced than is dissolved and therefore it accumulates. Bating consists of the reduction of plumping, separation of the fibres into fibrils, removal of degraded collagen, cleansing of the grain, production of silky grain, and a minimum hydrolysis of the collagen itself.

D. WOODROFFE.

Tannin content of British Columbian *Alnus rubra*. R. H. CLARK and H. R. OFFORD (Trans. Roy. Soc. Canada, 1926, [iii.], 20, III., 149—152).—The variation of the tannin content of the dried bark of red-alder from the Vancouver district with the season of the year has been investigated. During October and November the tannin content is of the order 5—6%, falling to below 3% in January, and rising again to 6—7% in March. The tannin content does not warrant commercial extraction.

J. S. CARTER.

Electrometric determination of hydrogen-ion concentration in colloidal solutions, particularly tan liquors. V. KUBELKA and J. WAGNER (Collegium, 1926, 266—269).—The errors obtained in determinations of hydrogen-ion concentration owing to the foaming caused by organic colloids are overcome by the Kubelka–Belavsky form of electrode (Collegium, 1925, 247). The ordinary electrode becomes coated with an impermeable membrane of coagulated colloidal particles which cannot be removed by washing. The repeated platinising of the electrode is inconvenient and interferes with the measurements. In the Kubelka–Belavsky electrode the platinum wire is adjustable and the amount dipping into the liquor can be regulated, starting with a small portion and increasing the depth of immersion so as to expose a fresh surface after each measurement. Comparative results are given to show the differences obtained with the new electrode compared with the old fixed type. One electrode of the new type will serve for 20—30 determinations before it needs to be re-platinised.

D. WOODROFFE.

Determination of chromium in used chrome [tanning] liquors. V. KUBELKA and J. WAGNER (Collegium, 1926, 257—266).—The chrome liquor is filtered and diluted to a content of 1% Cr. 50 c.c. of the diluted liquor are pipetted into a 500-c.c. graduated flask, potassium hydroxide is added until the chromium hydroxide precipitated at first re-dissolves, and then a further 1 c.c. of 25% potassium hydroxide added. The clear chromite solution thus obtained is heated for about 10 min. on the water bath or over a small flame. As soon as the solution becomes bright yellow, N-potassium permanganate is run in drop by drop until a brown flocculent precipitate of manganese dioxide is produced, the mixture is well shaken until effervescence ceases, made up to the mark after 5 min., and the chromium determined in 100 c.c. of this solution iodometrically.

D. WOODROFFE.

Examination of leather. G. POVARNIN and J. SCHICHIREV (Collegium, 1926, 269—275).—In the determination of soluble matter extracted from leather by alcohol and water, more tannin is extracted if the treatment with alcohol precedes that with water. The higher the "true tanning figure" (cf. B., 1923, 1141 A; 1926, 556) the greater is the wearing power of the dried leather. Wearing tests with sand are done better by rotating the samples of leather on sand than by means of a falling stream of sand. The wearing properties change inversely with the tensile strength. In estimating the effect of free sulphuric acid on leather, the amount of soluble salts in the leather must also be taken into account. A fresh variable is suggested for judging the regularity of the tannage. The leather is cut into three layers of equal thickness. The nitrogen content of each layer is determined and also that of the original unsplit leather. If N_u, N_m, and N_l are the nitrogen contents of the upper, middle, and lower layers respectively, and A that of the original leather, then $100\,[1-(N_m-N_o]/A)$ and $100\,[1-(N_m-N_l)/A]$ are the "coefficients of regularity of tannage" for the grain and flesh sides respectively of the leather. In spite of the higher regularity of tannage, the "true tanning figure" of drum-tanned leather is lower than that of pit-tanned. The wearing properties of the drum-tanned leather are greater than those of pit-tanned in spite of a higher content of free sulphuric acid. The pit-tanned leather is submitted to a swelling before tannage and from previous researches the authors have shown that such swelling is detrimental to the wearing properties.

D. WOODROFFE.

PATENTS.

Bleaching sole leather. J. RAISOR (U.S.P. 1,588,686, 15.6.26. Appl., 25.1.22).—Leather is dipped successively into (1) water at approximately 49°, (2) an alkali solution, (3) an aqueous solution, *d* 1·383, containing equal pts. by wt. of sulphuric acid, sodium chloride, and alum, (4) dilute sulphuric acid, *d* 1·162, (5) water.

L. A. COLES.

Recovery of chromium [from leather waste]. ELLENBERGER & SCHRECKER (G.P. 427,807, 29.3.24).—Leather waste from which glue has been extracted is treated hot with chlorine to obtain chromic chloride.

A. R. POWELL.

Dressing skins. A. J. CLERMONTEL (E.P. 250,178, 15.1.26. Conv., 31.3.25).

XVI.—AGRICULTURE.

Some relations between climate and soils in Europe. A. MEYER (Chem. Erde, 1926, 2, 209—347).—An exhaustive account of investigations on the influence of climatic factors, particularly temperature and abundance or shortage of water, on the origin and characters of European soils. A bibliography of 160 titles is included. C. T. GIMINGHAM.

Crust soils, crust formation, and red soils, with special reference to the soil formations of Palestine. E. BLANCK, S. PASSARGE, and A. RIESER (Chem. Erde, 1926, 2, 348—395; cf. B., 1925, 20).—A discussion of the occurrence and formation of crusts and crust soils, particularly chalk crusts and red soils, is followed by an account of the distribution of soil types in the hill country of Palestine and of the investigation of some characteristic soil profiles. C. T. GIMINGHAM.

Soil microbiology. II. Nitrogen-fixing bacteria. S. WINOGRADSKY (Ann. Inst. Pasteur, 1926, 40, 455—520; cf. B., 1925, 328, 465; 1926, 415, 505).—The author's methods for classifying soils according to their nitrogen-fixing powers are described and a detailed account is given of the experiments on which the procedures are based. C. T. GIMINGHAM.

Nature and availability of the plant-food constituents of Philippine guano. M. TIRONA (Philippine J. Sci., 1926, 30, 69—78).—The phosphorus in Philippine guanos is generally of rather low availability and exists mainly as phosphates of iron and aluminium. The total nitrogen present ranges from 0·7 to 4·7%, and a high proportion (16—60%) is soluble in water. About one quarter of the soluble nitrogen is in the form of nitrates, a feature which distinguishes Philippine guanos from those found elsewhere. Potassium is always present but the amount seldom exceeds 1·5% K_2O. C. T. GIMINGHAM.

Determination of ammonia nitrogen in fertilisers by visual conductometric titrations. G. JANDER and O. PFUNDT (Z. angew. Chem., 1926, 39, 856—858).—Ammonia nitrogen may be determined by titration of the aqueous extract of fertilisers with sodium hydroxide, the course of the reaction being determined by a conductometric method in which the usual telephone is replaced by a crossed thermocouple combined with a galvanometer or millivoltmeter. The conductivity of the solution is proportional to the square root of the throw of the galvanometer, so that by plotting this against the c.c. of alkali added a series of intersecting straight lines is obtained. The first point of intersection corresponds with the alkali necessary to neutralise the free acidity, the second with that required to convert the primary phosphate into secondary, and the third with the liberation of the ammonia. The last-named result must be corrected for the precipitation of calcium phosphate by determining the calcium in a separate portion of the solution by precipitation as oxalate after addition of acetic acid. A. R. POWELL.

Determination of calcium carbide in calcium cyanamide. G. FLUSIN and H. GIRAN (Compt. rend., 1926, 182, 1628—1629).—The acetylene disengaged by treatment with boiling water is aspirated into ammoniacal silver nitrate solution. Treatment of the mixed precipitate of silver acetylide and silver sulphide with hydrochloric acid gives silver chloride and unchanged sulphide. The chloride is dissolved out with ammonia and determined by titration with potassium cyanide. M. CLARK.

Action of farmyard manure alone and in combination with mineral fertilisers. KLEBERGER (Z. Pflanz. Düng., 1926, B 5, 241—271).—The results of field experiments, extending over seven successive crops—potatoes, wheat, barley, green crop, sugar-beet, wheat, oats—are recorded, the use of dung with and without various combinations of mineral fertilisers being compared. In addition to the total yields of produce, the average amounts of nitrogen, potassium, and phosphorus taken up by the crops are given and the effects of the various treatments on the utilisation of plant nutrients are discussed. The results are also considered from the economic point of view. A combination of dung and complete mineral fertilisers gave in most cases the highest yields. C. T. GIMINGHAM.

Asahi-Promoloid. F. MÜNTER (Z. Pflanz. Düng., 1926, B 5, 272—274).—No increases of yield of lupins, carrots, and lettuce were obtained by the use of Asahi-Promoloid in vegetation experiments with a loamy soil. C. T. GIMINGHAM.

See also A., July, 673, **Adsorption and mobilisation of the potassium ion in colloidal clays** (DEMOLON). 679, **Anomalous flocculation of clay** (KERMACK and WILLIAMSON). 762, **Phosphates in vegetation experiments** (DOMONTOVICH).

Coli-aerogenes **group [of bacteria] in soil.** KOSER.—See XXIII.

Silicofluorides as insecticides. MARCOVITCH.—See XXIII.

PATENTS.

Fertilisers. W. R. FIELDING (E.P. 252,039, 16.12.24).—An organic material, such as straw, bracken, peat, sawdust, wood pulp, sewage sludge, or the like, is treated with a solution of sodium carbonate or sulphate or other efflorescent salt, and is then placed in a bath containing potassium carbonate, sodium nitrate, or other fertilising substances. The resulting mass, with or without additions of other materials to supply soil deficiencies, is dried and graded for use. It is claimed that the treatment with sodium carbonate enables the organic base to keep a fertiliser, in which deliquescent substances may be present, in a dry condition. C. T. GIMINGHAM.

Manufacture of fertilisers. E. L. PEASE (E.P. 253,291, 16.4.25).—A mineral or organic calcium phosphate is mixed with a porous material, such as peat, coarse coke dust, or sewage powder, and is treated with crude phosphoric acid. The mass is then used as an absorbent for ammonia, whereby a dry porous product containing a high proportion of readily available phosphorus and nitrogen is obtained. C. T. GIMINGHAM.

Electrometric determination of the acidity or alkalinity of soils, waters, solutions, and the like. M. TRÉNEL (G.P. 413,043, 16.7.24. Addn. to 399,410; B., 1926, 208).—The apparatus described in the chief patent is modified in that one electrode is placed in the comparison solution contained in a porous porcelain cell, the pores of which are sufficiently fine to prevent diffusion, and the other is placed directly in the vessel containing the solution under investigation to which the quinhydrone has been added. The cells used are preferably cylindrical and may consist entirely of porous porcelain or only the bottom or the lower portion may consist of this material.

A. R. POWELL.

Fungicide. W. MOORE and C. D. VREELAND (U.S.P. 1,587,977, 8.6.26. Appl., 30.3.25).—A fungicide, comprising an insoluble copper compound having an adsorbed positively charged ion, is prepared by precipitating the copper from a solution of copper sulphate with lime, and adding about 5% of calcium acetate to the precipitate.

C. T. GIMINGHAM.

Producing fertiliser and cattle feed (U.S.P. 1,587,101).—See XIX.

Bactericide and fungicide (U.S.P. 1,585,792).—See XXIII.

Fumigant (U.S.P. 1,586,175).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Determination of the ash content of raw sugar by the electrical conductivity method. L. KAYSER (Z. Ver. deuts. Zucker-Ind., 1926, 369—380).—Comparison of the electrical conductivity method with the incineration (sulphate) process shows the agreement to be satisfactory in most of the samples of raw beet sugars examined, but in other cases, particularly with after-product sugars, the differences between the two processes were much beyond the limit of error of the latter method. Where they cannot be accounted for by the presence of insoluble matter in the sugars examined, these differences are due to variations in the composition of the mineral matter. J. P. OGILVIE.

Complete analysis of mixtures of starch sugars and cane sugar products. D. R. NANJI and R. G. L. BEAZELEY (J.S.C.I., 1926, **45**, 220—221 T).—A method is described for the determination of the chief constituents—dextrose, lævulose, sucrose, maltose, β-disaccharides and dextrin—which are present in mixtures of starch sugars and cane sugar products. Adulteration of a cane sugar product such as invert sugar with starch sugars may be detected qualitatively by the decrease in specific rotatory power after hydrolysis with 8% (by weight) sulphuric acid for 3 hrs. in a boiling water-bath, and by the presence of a considerable quantity of dextrorotatory matter unfermentable by Frohberg yeast. The quantitative method consists of the following operations:—(1) The reducing power of the sample is determined with Fehling's solution, by the volumetric method, and calculated in terms of apparent dextrose and apparent invert sugar. This reducing power represents the dextrose, lævulose, maltose together with the other reducing disaccharides, and dextrins present in the product. (2) The reducing power is then determined with Fehling's solution after inversion with 10% citric acid. The reducing power after inversion is calculated in terms of apparent invert sugar. It represents dextrose, lævulose, maltose, dextrin, and the invert sugar formed from sucrose present in the sample. (3) The reducing power of the sample is also determined iodometrically, and calculated in terms of apparent dextrose. Since iodine does not act on lævulose, the reducing power by this method represents all the reducing sugars in the sample except lævulose. In practice a slight correction has always to be applied for the very small absorption of iodine by lævulose. (4) The rotation of a 10% solution of the sample is determined, before and after fermentation with Frohberg yeast. (5) The specific gravity of a 10% solution is determined before and after fermentation with Frohberg yeast. In the latter case the alcohol is, of course, removed before taking the gravity. (6) Lastly, ash and nitrogen in the sample are determined in the usual way. From these data it is possible to determine the amounts of the various constituents present. An example is given showing the method of calculation.

Hydrolysis of starch by acids. D. R. NANJI and R. G. L. BEAZELEY (J.S.C.I., 1926, **45**, 215—219 T).—A fraction resembling *iso*maltose is present among the products of the hydrolysis of starch by acids only after 78% of the starch has been hydrolysed to maltose and dextrose. This fraction is unfermentable by low attenuating yeasts, and its specific rotatory power varies ($[\alpha]_D$ 55—81°) according to the stage of hydrolysis. It yields a crystalline osazone, m.p. 155—160°, resembling *iso*maltosazone. The specific rotatory power of different osazone preparations varies considerably, some being dextrorotatory ($[\alpha]_D$ +15—20°), whilst others are lævorotatory ($[\alpha]_D$—11°). It is probable, therefore, that the fraction is not a homogeneous substance, but a mixture of gentiobiose formed by reversion, and true *iso*maltose formed directly from the starch. The hydrolysis of starch by acids appears to be the resultant of over a dozen reactions taking place simultaneously. The liquefaction observed during the earliest stages of hydrolysis is shown to be due to the conversion of the calcium salts of the esters of

amylose and amylopectin into the free phosphoric esters. The whole of the phosphorus in starches is present in organic combination and liquefaction of starch takes place before the hydrolysis of the phosphoric esters. A method is described for studying quantitatively the products obtained at different stages of hydrolysis. The percentages of dextrin and the *iso*maltose fraction are determined by fermentation with Saaz and Frohberg yeasts, both being unfermented by the former, and dextrin only by the latter. The optical rotation of a 10% solution is taken before and after fermentation with Saaz yeast. The reading of the solution before fermentation is due to dextrin, *iso*maltose, maltose, and dextrose. The reading of the same solution after fermentation is the rotation due to dextrin and *iso*maltose. By subtracting the percentages of dextrin and *iso*maltose, together with the percentages of ash and proteins from 100, the percentage of maltose and dextrose together is obtained. If the specific rotation of the mixture of these two sugars then be calculated, the relative proportions of the two sugars can be determined by the equation $52{\cdot}5x+138(1-x)=[\alpha]_D$ of the mixture of dextrose and maltose, where $x =$ the amount of dextrose in 1 g. of the mixture.

Comparison of the hydrolytic [liquefying] action of various substances on starch. R. HALLER and A. HOHMANN (Textilber., 1926, 7, 239—242).—The effect on starch of various oxidising agents and enzymes was determined by exposing gelatinous solutions of potato starch to the action of the various agents at 65° under specified conditions, subsequently terminating the action of the agents by heating for 10 min. at the boiling point, and then comparing the viscosities of the products at 70° in an Ostwald viscosimeter. The liquefied starch products obtained by the action of enzymes are less viscous than those obtained by means of oxidising agents. The liquefying actions of oxidising agents and enzymes are largely dependent on their concentrations. The liquefying action of Aktivin on starch in glass vessels is assisted by the presence of traces of copper sulphate and nickel sulphate, but not by palladium chloride. The viscosities of the products obtained by the action of concentrations of Aktivin, bleaching powder, and sodium perborate proportional to their oxidising powers are in the ratio 2·70 : 3·58 : 6·50. Less viscous starch products are obtained by the action of Aktivin in the presence of sodium carbonate. The liquefied products obtained by means of Aktivin, bleaching powder, sodium perborate, Novo-Fermasol, Diastafor extra, Diastaphor double conc., Biolase, and Degomma D gave blue, blue, blue, bluish-red, red, red, yellow, violet, and blue colours respectively, with an alcoholic solution of iodine, and contained (as determined by a method of dialysis under similar conditions) 0%, 0%, 0%, 4·83%, 8·15%, 15·76%, 16·64%, 11·88% and 31·50% of reducing sugars (calc. as dextrose on the weight of starch used), respectively. The products obtained by means of Aktivin in the presence of copper sulphate (as catalyst) are whiter than the products similarly obtained by means of Aktivin in the presence of sodium carbonate, or by sodium perborate or bleaching powder, and are therefore more satisfactory for the dressing of textile materials. A. J. HALL.

Unsaturated fatty acids associated with corn [maize] starch. T. C. TAYLOR and L. LEHRMANN (J. Amer. Chem. Soc., 1926, 48, 1739—1743).—The fatty acids liberated on hydrolysis of maize starch (α-amylose) have been identified as palmitic acid (24%), oleic acid (40%), and linoleic acid (36%). Nitrogen and phosphorus are absent.
F. G. WILLSON.

See also A., July, 714, *iso*Maltose (ISAJEV). 715, **Starch, Amylobiose** (PRINGSHEIM and STEINGROEVER).

Beet molasses as raw material for yeast production. CLAASSEN.—See XVIII.

PATENTS.

Process of treating sugar-beet diffusion juice. H. S. THATCHER and S. E. JOSI, Assrs. to CELITE Co. (U.S.P. 1,586,486, 25.5.26. Appl., 22.8.23).—Beet diffusion juice is heated and passed through a coating of diatomaceous earth previously formed on a filtering surface by passing through the filtering apparatus a suspension of the earth in purified beet juice.
J. P. OGILVIE.

Calandria for evaporators of sugar mills. A. P. LEONARD (U.S.P. 1,586,814, 1.6.26. Appl., 17.3.24).—A calandria has a closed heating chamber with downtake and steam inlet, a baffle forming a steam passage leading from the inlet, and an outlet for the passage at the end opposite the inlet. A number of circulation pipes are provided in the chamber between the inlet and outlet, these being so arranged as to form an unobstructed passage-way from the inlet along the baffle. J. P. OGILVIE.

XVIII.—FERMENTATION INDUSTRIES.

Beet molasses as raw material for the production of yeast by the aeration process. H. CLAASSEN (Z. Ver. deuts. Zucker-Ind., 1926, 349—368; Z. angew. Chem., 1926, 39, 880).—Using final molasses from German beet factories and refineries containing 1·2—1·9% N, white, vigorous bakers' yeast, conforming to standard fermentation tests, and of good keeping quality, was produced in small-scale tests. Phosphoric acid was the only nutrient added. Yields of yeast (25% of dry substance) equal to 41—59% of the molasses were obtained, and the amount of nitrogen assimilated by the yeast varied from 40 to 60% of that originally present. J. P. OGILVIE.

Buffer substances in wort and beer. II. P. KOLBACH (Woch. Brau., 1926, 43, 277—280, 289—294).—This is mainly a survey of the subject. The advantages of a plentiful supply of buffer substances in malt liquors outweigh any disadvantages. There is conclusive evidence that

acid flavour is not determined solely by the concentration of hydrogen ions. Beer has a much less pronounced acid flavour than would be anticipated from its p_H value, probably owing to the mollifying influence of proteins (cf. Bermann, B., 1925, 1006). For practical purposes the buffering characters of malt extracts and beers can be satisfactorily estimated by titration in presence of different indicators, *e.g.*, neutral-red and γ-dinitrophenol (see Windisch and others, B., 1925, 951). Windisch has shown that the effect of phosphates can be approximately estimated by titration in presence of two indicators, before and after precipitation with barium hydroxide. The only other inorganic buffer substance in malt is silicic acid, but it is too small in amount to influence the reaction of wort or beer. It deserves investigation, however, in relation to turbidity in beer, for it appears to be present in an unstable condition, since it constitutes about 25% of the total mineral matter present in the coagulum formed when beer is heated.

J. H. LANE.

Influence of hop constituents on head-formation in beer. W. WINDISCH, P. KOLBACH, and W. BANHOLZER (Woch. Brau., 1926, 43, 207—209, 217—223, 229—235, 241—246, 253—258).—Of the constituents of beer, those derived from the hops contribute most to head-formation and head-retention. The length of time during which wort is boiled with hops has little influence on the head-forming power of the fermented beer; the authors found no difference after boiling for 1 and 2 hrs., respectively, but boiling for 3 hrs. produced a slight decrease in head-forming power. Within the practical range of hydrogen-ion concentration in beer head-formation is impaired by an increase of acid reaction, owing doubtless to lessened solubility of the foam-forming hop constituents. In beer made from unhopped wort, change of reaction does not influence head-formation. Ether will extract from hops the whole of the foam-forming constituents. The most important of these is the α-bitter acid, humulone, which has also been shown to be the most important in respect of flavouring and antiseptic properties. It is about 2·5—5 times as active as the β-bitter acid, lupulone, in promoting head-formation, and as it is present in hops in larger quantity than the latter, the foam-forming properties of different samples of hops show a much closer correspondence with their contents of humulone than with their contents of total resins. The soft resins into which humulone is partially converted during wort-boiling also have an important head-promoting influence on the fermented beer. Humulinic acid, a product of hydrolysis of humulone, is even more active than humulone itself in this respect. The method used for measuring head-forming and head-retaining power was that recently described (B., 1925, 1007). J. H. LANE.

Fermentation products from cellulose. H. B. SPEAKMAN (Pulp and Paper Mag., 1926, 24, 731—736).—The fermentation of sugars such as galactose and xylose by the butyl alcohol–acetone fermentation organisms depends on their initial concentration, and upon the proportion of fermentable sugars with which they are mixed. Thus, a 2% solution of xylose is completely fermented, whereas a 5% solution remains unchanged; galactose alone is fermented to the extent of 10%, but this is increased to 50% in the presence of an equal amount of dextrose. The yield of acetone by the fermentation of sulphite-cellulose waste liquor is increased if this is neutralised, and if the maize mash culture is allowed to develop for 12—24 hrs. before the addition of the sulphite-cellulose waste liquor. A. GEAKE.

Determination of citric acid and the results of experiments with wine. O. REICHARD (Z. Unters. Lebensm., 1926, 51, 274—289).—Methods for the determination of citric acid are reviewed. The question as to how far citric acid can be described as a natural constituent of wine, and the possibility of distinguishing natural citric acid and that artificially added are discussed. Denigès' method for determining citric acid (cf. B., 1898, 802) is not satisfactory since other substances, *e.g.*, malic acid, produce a turbidity with the mercuric sulphate solution. Stahre's reaction as modified by Kunz (B., 1915, 974; cf. Von der Heide and Straube, B., 1922, 912 A) gives a trustworthy method for the detection and the determination of citric acid in wines.

A. G. POLLARD.

Pyrogenic dehydration of fusel oil. M. GIUA and L. THUMIGER (Atti R. Accad. Sci. Torino, 1926, 61, 199—208; Chem. Zentr., 1926, I., 3508).—Fusel oil obtained by fermentation by the Fernbach process contains about 65% of *n*-butyl alcohol, whereas ordinary fusel oil contains about 24% of *iso*butyl alcohol, 68% of amyl alcohol, 7% of propyl alcohol, and small quantities of esters, furfuraldehyde, and other compounds. On passing fusel oil vapour mixed with ethyl alcohol over heated catalysts, mixtures of ethyl esters of high boiling point, suitable, *e.g.*, for denaturing alcohol–benzine mixtures, are obtained. Olefine hydrocarbons of high boiling point are obtained by passing fusel oil vapour over heated mixtures of alumina and chromic oxide.

L. A. COLES.

See also A., July, 715, **Specific action of amylases** (PRINGSHEIM and LEIBOWITZ); **Fermentative degradation of starch by "biolase"** (PRINGSHEIM and SCHAPIRO), 757, **Methods for measuring rate of hydrolysis of starch and dextrin by taka-diastase. Effect of p_H on starch-liquefying and dextrin-liquefying powers of taka-diastase** (MASLOW and DAVISON); **Enzymes and light. Diastase** (PINOUSSEN). 758, **Action of drugs and irradiation on yeast** (ZELLER); **Nitrogenous equilibrium in the yeast cell and augmentation of the invertase action** (VON EULER, JOSEPHSON, and FINK). 759, **Yeast amylase and fermentation of polysaccharides** (GOTTSCHALK); **Proteases of yeast** (WILLSTÄTTER and GRASSMANN); **Forms of lactic acid produced by pure and mixed cultures of bacteria** (PEDERSON, PETERSON, and FRED). 764, **Determination of volatile fatty acids in bacterial cultures** (VIRTANEN).

Dry yeast. ULEX.—See XIX.

PATENTS.

Absolute alcohol process. E. I. CLAPP, Assr. to U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,586,732, 1.6.26. Appl., 18.11.24).—In the manufacture of absolute alcohol by fractional distillation of a mixture of alcohol, water, and low-boiling hydrocarbon in a column still, the still is supplied with a mixture of the three substances in which the ratio of the low-boiling hydrocarbon to water is between 10·5 : 1 and 17 : 1. T. H. POPE.

Enzymes and process of isolating them from their solutions. H. ALTGELT and O. HOCHMUTH, Assrs. to KALLE & CO. A.-G. (U.S.P. 1,590,663, 29.6.26. Appl., 17.3.25. Conv., 17.3.24).—See E.P. 251,405 ; B., 1926, 604.

Centrifugal liquid purifiers (E.P. 252,421).—See I.

Producing fertiliser and cattle feed (U.S.P. 1,587,101).—See XIX.

XIX.—FOODS.

Gluten. B. d'ARBOUET (Rev. gén. Colloid., 1926, **4**, 97—107).—The colloidal nature of gluten and its importance in bread-making is discussed. Differences in the baking values of flours are largely explained by the physical condition of the gluten. The presence of a continuous network of colloidal gluten in the dough is important for good baking properties. Inherent differences in the grain, methods of grinding, and the reaction of the dough are the main factors affecting the colloidal condition and "cohesiveness" of the gluten. The poor baking value of rye flour and the effect of small additions of pea flour to a weak wheat flour are explained on similar lines. A. G. POLLARD.

Preparation and examination of cheese fat. O. BAUMANN (Z. Unters. Lebensm., 1926, **51**, 267—272).—For the isolation of cheese fat the process of Grossfeld (extraction with trichloroethylene after boiling with hydrochloric acid) gave the highest yield. Fats obtained from a number of cheese samples by neutral, acid, and alkaline processes of extraction were examined. Excepting in the case of the Reichert–Meissl values, no definite relationship was found between the process of extraction and the standard analytical values determined in fat analysis. Reichert–Meissl values were highest where the fats were extracted by the neutral process and lowest where the acid process was used. In no case, however, was the value lower than that for butter-fat. A. G. POLLARD.

Determination of starch in pectin and apple juices by a sedimentation process. H. ECKART and A. DIEM (Z. Unters. Lebensm., 1926, **51**, 272—275).—Ten c.c. of the sample are heated in a water bath for 10 min. with 80 c.c. of calcium chloride solution (1 pt. of calcium chloride to 2 pts. of water). The latter should be made neutral to phenolphthalein before use. The mixture is transferred to a graduated flask and diluted to 100 c.c. with calcium chloride solution. A standard starch solution is prepared by heating on a water bath 0·1 g. of pure potato starch with a few drops of calcium chloride solution and adding gradually 50 c.c. of water. The solution is then diluted to 100 c.c. ; 10 c.c. (or suitable dilutions) of the two solutions are treated with excess of iodine in potassium iodide solution in graduated centrifuge tubes. After 30 min., when the precipitates have flocculated, the solutions are centrifuged and the starch contents compared by means of the volumes of the precipitates. A. G. POLLARD.

Determination of shell in cocoa and chocolate. J. GROSSFELD (Z. Unters. Lebensm., 1926, **51**, 249—262).—Moderate quantities of shell in cocoa powders may be detected by determining the crude fibre and the ratio of nitrogenous substances to crude fibre. This ratio is greater than 4 for cocoa and approximately 1 for shell. The silica content also gives an indication of the presence of shell. The Filsinger sedimentation process (Z. öffentl. Chem., 1899, **5**, 27) is useless for modern cocoa powders. It is shown that during the treatment with water the proportion of crude fibre in the residues increases and the nitrogen content decreases. The difference in the crude fibre content of cocoa and shell is lessened by leaching with water, but the actual values tend to become more constant. A process is described whereby the proportion of shell in cocoa powders may be calculated from the percentage residues after sedimentation and the crude fibre content of these residues. A. G. POLLARD.

Incineration [of foodstuffs]. P. FORTNER (Z. Unters. Lebensm., 1926, **51**, 300—301).—In the incineration of residues containing much sugar preliminary heating at low temperatures hastens the process. After evaporation on a water bath the residue is transferred to a platinum crucible which is placed on an asbestos plate heated by a burner placed 3—4 cm. from the crucible until the smell of burnt sugar ceases, when the flame is brought steadily nearer and increased. Finally the crucible is heated over the naked flame. To avoid loss of material by spitting during the ashing of foodstuffs containing much water, fat, or common salt, the residue after evaporation is covered with a circle of filter paper, cut to fit the crucible. If this is pressed down on the material it absorbs the fat and the subsequent ignition can be carried out without loss. A. G. POLLARD.

Can corrosion and blackening in certain marine products. D. B. DILL and P. B. CLARK (Ind. Eng. Chem., 1926, **18**, 560—563).—Free oxygen and volatile bases were not found to be significant factors in can corrosion by canned marine products. The content of sulphide sulphur is related to the blackening which is increased by rise of temperature. Of all the marine products examined only *Crustacea* contained organic sulphur metastable at ordinary temperatures and only *Crustacea* blackened to any great extent. The conversion of

organic sulphur into sulphide sulphur is accelerated by the metal of the container, and the proportion of sulphide sulphur increases during storage. All products which blacken and corrode the container were found to be on the alkaline side of p_H 6·5. Products more acid than p_H 6·5 do not blacken and for the most part do not corrode the container. D. G. HEWER.

"Blown" tins. An exceptional case. T. B. SHAW and R. C. FREDERICK (J. Roy. Naval Med. Service, April, 1926, Reprint).—The occurrence of "blown" tins is an indication of the unwholesomeness of a tinned food, but this rule does not apply to tinned coffee; "blowing" in the latter case may be due to a normal reaction between the freshly ground coffee and atmospheric oxygen. C. O. HARVEY.

Dry yeast. H. ULEX (Chem.-Ztg., 1926, **50**, 475—476).—Used as fodder, dry yeast is put on the market in unextracted and extracted forms, which vary in appearance, taste, fat and protein contents, and in the amount of aqueous extractives. The unextracted form, which is more expensive, produces more rapid increase of weight and improved growth in the animal. Its superiority depends upon the content of the water-soluble substances, which are absent from the extracted variety, having been removed during the process of manufacture. In this process of preparation, no fat is lost from the yeast and owing to the removal of the soluble substances, the fat content of the extracted variety is correspondingly higher. On an average, unextracted yeast contains 48·8% of protein, 4·1% of fat, and yields approximately 36% of extractives. The corresponding values for the extracted form are 34·9%, 6·6%, and 16%. The contents of phosphoric acid, sodium chloride, and the alcoholic extract are almost identical in the two varieties. C. RANKEN.

See also A., July, 760, **Concentration of growth-promoting principle (vitamin-*B*) obtained from yeast** (LEVENE and VAN DER HOEVEN). 761, **Mineral composition of sunflowers grown for silage** (NEIDIG); **Proteins of green-forage plants** (DAVIES). 762, **Nutritive value of various layers of the wheat and maize kernel** (KLEIN, HARROW, PINE, and FUNK); **Biological value of bread protein** (HINDHEDE).

Detection of tallow in lard. MUSCHTER and VISSER.—See XII.

Determining milk fat in mixtures of fats. GROSSFELD.—See XII.

Crude fibre determinations in spices. OTTE and WEISS.—See XX.

PATENTS.

Food preparations made from meat and like edible animal matter. D. THOMSON and F. N. PICKETT (E.P. 252,754, 8.12.24).—The minced meat, mixed with water, is passed through two high-speed disintegrating machines until a practically colloidal suspension of the meat in water is produced. The acid liquid is neutralized by the addition of sodium hydroxide and the liquids remaining are removed by treatment in a centrifuge or by filtering. The product is readily assimilable and may be administered as a medicinal food in admixture with alcohol. E. H. SHARPLES.

Producing fertiliser and cattle feed. A. WOOLNER, JUN. (U.S.P. 1,587,101, 1.6.26. Appl., 26.12.24).—The density of thin butyl alcohol distillation slop is raised by the addition of material of relatively high density containing sugar, so that the valuable constituents of the slop can be economically utilised in the production of fertilisers and feeding-stuffs. L. A. COLES.

Manufacture of bread. H. HEWITT, Assr. to BRIT. ARKADY CO., LTD. (U.S.P. 1,589,311, 15.6.26. Appl., 19.8.24. Conv., 12.12.23).—See E.P. 232,284; B., 1925, 564.

Preparation of cheese having an increased or supplemented vitamin content. H. LIEBERS, Assr. to HAMBURGER & CO. GES.m.b.H. (U.S.P, 1,590,837, 29.6.26. Appl., 9.4.24. Conv., 21.12.23).—See E.P. 226,549; B., 1926, 460.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Alkaloidal content of British Columbian *Datura stramonium* and *Conium maculatum*. R. H. CLARK and H. R. OFFORD (Trans. Roy. Soc. Canada, 1926, [iii.], 20, III., 153—155).—The dried seeds and stems of *Datura stramonium* contain about 0·28% and 0·06% respectively, of alkaloids, mainly atropine, hyoscyamine, and hyoscine. The dried leaves contain about 0·3% of alkaloids, the amount rising to about 0·4% during August, when the plant is in flower. *Conium maculatum* (spotted hemlock) grows to much greater heights in British Columbia than in Great Britain and the coniine content is considerably above the average found elsewhere. The dried seeds contain about 0·92% of alkaloids, and the dried stems about 0·025%. J. S. CARTER.

Piperonal in vanilla extract. C. B. GNADINGER (Ind. Eng. Chem., 1926, **18**, 588—589).—Piperonal could not be detected in any variety of vanilla beans, except Tahiti beans and vanillons, which appear to contain very small quantities. In using the gallic acid test for piperonal it is necessary to separate the aldehydes from any anisyl alcohol present (cf. B., 1925, 376), as the deep red colour produced by the latter will mask the blue colour of the reaction. D. G. HEWER.

Manufacture of phenacetin from *p*-chloronitrobenzene. D. H. RICHARDSON (J.S.C.I., 1926, **45**, 200—203 T).—By heating *p*-chloronitrobenzene at 60° for 140 hrs. in a 0·5 *N*-solution of caustic soda in 95% alcohol free from acetaldehyde, 92% of the theoretical yield of *p*-nitrophenetole may be obtained, the only by-product being *p*-nitrophenol. *p*-Nitrophenetole may be converted directly into phenacetin (96% yield) by refluxing with iron filings and five

times its weight of glacial acetic acid, but even when allowance is made for the value of the acetic acid recovered this method is more costly than the reduction of nitrophenetole to phenetidine and the acetylation of the base. In material cost it is cheaper to reduce nitrophenetole with iron and hydrochloric acid than with sodium sulphide, but the latter process has the advantage in ease of working and isolation of the product. Various methods of isolating the phenetidine are compared, but none yielded more than 75% of the theoretical amount. Both phenetidine base and hydrochloride can be satisfactorily acetylated by distilling with a solution of acetic acid in toluene, the distillate being dehydrated by anhydrous sodium acetate and returned to the reaction vessel.

Acidimetric titration and composition of commercial lactic acid. R. EDER and F. KUTTER (Helv. Chim. Acta, 1926, **9**, 557—578; cf. A, 1926, 499).—A critical survey of the literature on the formation and determination of lactic acid and its dehydration products is given. In the absence of lactide the following method may be used for the determination of free lactic acid and lactyl-lactic acid in commercial lactic acid. About 0·4 g. of concentrated lactic acid (p) is diluted with 20 c.c. of water, and titrated directly with 0·1 N-sodium hydroxide (a) using neutral-red as an indicator. Sufficient 0·1 N-alkali (b) is then added to hydrolyse the lactyl-lactic acid, the solution heated for 10 min. on the water bath, and titrated with 0·1N-acid to about 1—2 c.c. excess (c). After again warming, the excess of acid is titrated with standard alkali (d). Since 1 c.c. of N-sodium hydroxide solution=0·09005 g. of lactic acid, free acid present$=0{\cdot}9\,(a+c-b-d)/p\%$, and the lactyl-lactic acid$=1{\cdot}8\,(b+d-c)/p\%$. Direct titration of lactide with N-alkali solution determines 50% of the lactide in accordance with the equation

$$C_6H_8O_4+NaOH=CHMe(OH){\cdot}CO{\cdot}O{\cdot}CHMe{\cdot}CO_2Na$$

but it is not attacked by an alcoholic solution; and hence the following method is evolved for the determination of free lactic acid ($X\%$), lactyl-lactic acid ($Y\%$), lactide ($Z\%$), and water ($W\%$) in the presence of each other. Direct titration with N-sodium hydroxide solution in aqueous solution, using phenolphthalein as an incidator (a), gives the value of $X+Y/2+Z/2$, and subsequent hydrolysis with N-alkali in aqueous solution as described above (b) represents the value of $Y/2+Z/2$. Direct titration of the sample dissolved in absolute alcohol with N-alcoholic sodium hydroxide, using thymol-blue as an indicator, represents $X+Y/2$, and subsequent hydrolysis for 20 min. on the water bath after the addition of 25 c.c. of water represents $Y/2+Z$. The water content is found by difference. Analysis of samples of 90% and 100% commercial lactic acid showed that the quantity of lactide present is less than the value of the experimental error, and hence commercial lactic acid contains only free lactic acid, lactyl-lactic acid, and water.

J. W. BAKER.

Electrolytic preparation of glycollic acid. V. TOJA and U. CEVA (Giorn. Chim. Ind. Appl., 1926, 8, 3—7).—The most economical method of preparing glycollic acid consists in the electrolytic reduction of oxalic acid in presence of sulphuric acid, using pure lead cathodes and comparatively low current densities. The sulphuric acid is removed by treating the liquor with lime, and the solution thus purified is concentrated in a vacuum and the resulting syrup allowed to cool. The acid then deposited is of good quality. An outline is given of an industrial plant and the view is expressed that the manufacture of the acid must be conducted in conjunction with that of oxalic acid. At present, however, glycollic acid can scarcely compete seriously with tartaric acid for industrial purposes. T. H. POPE.

Industrial extraction of citric acid from lemon juice. B. MELIS (Annali Chim. Appl., 1926, **16**, 135—141).—To extract pure calcium citrate from lemon juice, the juice is filtered after being mixed with one or more of the following: kieselguhr, tufa, kaolin, talc, or natural amorphous or precipitated gypsum, particularly that obtained by the decomposition of calcium citrate by sulphuric acid. The ratio of filtering material to juice should not exceed 1:5 if filter-presses, or 1:10 if continuous, revolving, vacuum filters are used. The filtered juice is neutralised, when boiling, with dense homogeneous milk of lime, free from magnesia; this addition is made in small amounts and with vigorous stirring. The final tenth of the total acidity may be neutralised by English chalk or by powdered marble. T. H. POPE.

Comparative crude fibre determinations in spices. W. OTTE and H. WEISS (Pharm. Zentr., 1926, **67**, 401—404).—The method of Huggenberg (Mitt. Lebensm. Hyg., 1916, **7**, 297) for the determination of crude fibre in spices is described and is recommended as a rapid "sorting" test, especially for pepper. Results given by 20 samples of pepper were, on an average, 0·78% lower than those given by the Henneberg and Stohmann method (Weender process), which must, however, always be used as a standard in doubtful cases. For pepper husks and scraps the results are considerably lower and for cardamom fruits, husks, and seeds higher, than those obtained by the standard method. The causes for these deviations are discussed.

E. H. SHARPLES.

Essential oil of the needles of *Pseudotsuga Douglasii*, Carr. E. ALINARI (Annali Chim. Appl., 1926, **16**, 185—190).—From an American sample of this essential oil Brandel and Sweet (Pharm. Rev., 1908, **26**, 326) isolated fractions with the odours of pinene and limonene and identified camphene and borneol. Schorger (B., 1914, 1170) showed that the terpene fraction of a Californian oil consisted mainly of α- and β-pinenes, together with a small proportion of limonene, camphene being absent; the heavier fractions contained borneol. In an oil prepared from needles of English growth Bennett (Perf. Ess. Oil Rec., 1920, **11**, 218) found limonene, dipentene, and a small amount of pinene, the principal component of the oil being geraniol. In view of the discordance between these results, the author has analysed an oil

prepared from the needles of this tree grown in the forest of Vallombrosa. The greenish-yellow oil of pleasant aromatic odour has d^{15} 0·8712, $[\alpha]_D$ —25·43°, n_D^{20} 1·4736, ester value 18·87 (6·60% of bornyl acetate), ester value of acetylated oil 52·29 (about 9·94% of free alcohols in original oil); it consists mainly of β-pinene and of geraniol and nerol, which are mostly free but partly esterified by acetic and capric acids.

T. H. POPE.

Mentha pulegium [pennyroyal] oil. P. LIOTTA (Riv. Ital. Ess. Profumi, 1926, 8, 44—45; Chem. Zentr., 1926, I., 3507).—Steam distillation of 100 kg. of *Mentha pulegium* yielded 350—500 g. of a dirty straw-yellow oil having d 0·8400, α^{15} 26° 30′, and acid value 0·31. The oil is soluble in 2 pts. of 70% alcohol. and contains 0·85% of esters and 62% of pulegone and piperitone. L. A. COLES.

Essential oil from Medang Lesoh [*Cinnamomum Parthenoxylon*]. B. J. EATON and G. L. TEIK (Malay. Agric. J., 1926, 14, 81—83).—The oil is very similar to that formerly described (B., 1926, 215), consisting principally of safrole and having $d_{15\cdot5}^{15\cdot5}$ 1·101, n_D^{20} 1·538, ester value 2·5, and ester value after acetylation 8·4. It is optically inactive and is soluble in 3·3 vols. of 90% alcohol. E. H. SHARPLES.

See also A., July, 671, **Azeotropic properties of formic and acetic esters of saturated aliphatic alcohols** (HANNOTTE). 690, **Rate of reaction between ethylene and chlorine** (STEWART and FOWLER). 702, **Is Kjeldahl's method for determination of nitrogen in organic substances generally applicable?** (KÜRSCHNER). 722, **Synthesis of adrenaline. Preparation of pyrocatechol chloroacetates** (OTT); **Reaction of some polyhydric phenols with sodium antimonyl tartrate** (CHRISTIANSEN). 724, **Thyroxin. Constitution and synthesis of deiodothyroxin** (HARINGTON). 739, **Compound of veronal with pyramidone** (PFEIFFER and ANGERN). 744, **Alipine hydrochloride** (SEVILLA); **Alkaloid of *Julocroton montevidiensis*** (ANASTASI). 746, **Alkali xanthates as reagents for alkaloids** (NAVARRO); **Trypanocidal action and chemical constitution. Arylamides of aminohydroxyphenylarsinic acids** (HEWITT and KING). 749, **Refractometric determination of alcohols and esters in aqueous and in cottonseed oil solutions** (MUNCH). 750, **Electrometric determination of alkaloids without the use of the hydrogen electrode** (POPOV and MCHENRY). 755, **Preparation and chemical composition of active substances of *Digitalis* leaves** (CLOETTA).

Determination of citric acid. REICHARD.—See XVIII.

PATENTS.

Manufacture of colloidal organic mercury compounds soluble in water. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER & CO. (E.P. 243,361, 18.11.25).—Soluble colloidal organic mercury compounds, particularly suitable for immunising grain, are obtained by precipitating a solution of an organic mercury compound containing a protective colloid with an organic solvent which is miscible with water. The colloid may be added during the manufacture of the mercury compound, or the latter may be dissolved in an acid or alkali, and the colloid added before precipitation with the organic solvent. The precipitation is preferably effected from a neutral solution. Thus a solution of 10 pts. of the mercury compound of o-nitrophenol in 100 pts. of water and 2 pts. of sodium hydroxide is stirred into a solution of 100 pts. of albumose in 800 pts. of water. After neutralisation with acetic acid, precipitation is effected with acetone. The product contains 15·5% Hg and gives a neutral solution in water.

R. BRIGHTMAN.

Manufacture of aluminium ethylate [ethoxide]. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 245,473, 4.1.26. Conv., 3.1.25).—Alcohol containing a catalyst such as mercuric chloride, stannic chloride, or iodine in solution, is added slowly to a suspension of aluminium in aluminium ethoxide or in an inert solvent, such as xylene, the aluminium being in excess during the reaction. For example, 440 c.c. of alcohol containing in solution small quantities of mercuric chloride and iodine, are added slowly to a suspension of 100 g. of aluminium grit in 650 c.c. of boiling xylene. During the addition of about the first 320 c.c. of alcohol, the xylene is maintained at the boil by the heat of reaction, but external heat is applied during the addition of the remainder and for a further ¼ hr. after it has all been added. The mixture is filtered and distilled to remove mercury and xylene, yielding aluminium ethoxide readily soluble in the usual solvents, and containing no alcohol of crystallisation. L. A. COLES.

Isolating alcohols or phenols from mixtures. A. DEPPE SÖHNE, and F. O. ZEITSCHEL (E.P. 252,570, 21.7.25).—Mixtures containing alcohols or phenols are treated with quantities of the mixed anhydride of boric and acetic acids corresponding to the alcohol or phenol present. The volatile non-alcoholic constituents and the acetic acid formed are distilled off under reduced pressure, and the triborates of the phenols or alcohols which remain in the residue are hydrolysed by the addition of water or aqueous alkali. If solid the borates can be purified by recrystallisation prior to hydrolysis. In place of the boric-acetic anhydride may be used the corresponding double anhydrides of arsenious acid, antimonious acid, or phosphorous acid, and the acetic anhydride may similarly be replaced by any other organic acid anhydride. The reaction can also be effected with boric, arsenious, antimonious, or phosphorous acid or their anhydrides alone. The process may be applied to the preparation of pure menthol from Japanese peppermint oil, geraniol from Ceylon citronella oil, and linalool from shiu oil. Trial on a mixture of benzyl acetate and benzyl alcohol indicated that the method is quantitative. The method may also be applied to the approximate separation of primary, secondary, and tertiary alcohols, the reactivity of the three classes of alcohols being in the order named. Thus from a mixture of geraniol, borneol, and terpineol, almost

pure terpineol is readily obtained by heating with boric acid at 80—100° and hydrolysis of the borate formed. The arsenious acid anhydride method requires a somewhat higher temperature, *e.g.*, 150°.
R. BRIGHTMAN.

Process for catalytically preparing methanol [methyl alcohol] or higher alcohols or other oxygenated organic compounds or mixtures. L. CASALE (E.P. 252,573, 27.7.25).—Methyl alcohol or higher alcohols or other oxygenated compounds or mixtures of alcohols and other oxygenated compounds are obtained by catalytic reactions between carbon monoxide, carbon dioxide, hydrogen, and a gaseous hydrocarbon in a closed circuit of apparatus containing no moving parts. Circulation is obtained by admitting the mixture to the circuit at a higher pressure than that obtaining in the circuit, part of the potential energy of the mixture thereby being transformed into kinetic energy. In this way poisoning of the catalyst by the lubricating oil from the pistons and stuffing-boxes of circulating pumps is avoided.
R. BRIGHTMAN.

Preparation of ethylidene diacetate. SOC. CHIM. USINES DU RHÔNE (E.P. [A] 252,632, 30.11.25, and [B] 252,640, 6.1.26. Conv., 13.6. and 18.9.25).—(A) Ethylidene diacetate free from vinyl acetate is obtained in excellent yield by absorbing acetylene in acetic acid in the presence of the mercuric salt of a strong acid, such as sulphuric acid, sulphoacetic acid, benzene- or naphthalene-sulphonic acids, provided that a strong acid and an excess of acetic anhydride are present throughout. The free acid may be the same as or different from that giving the mercuric salt. The reaction is carried out at 80—90° until about 80% of the theoretical amount of acetylene has been absorbed by the acetic acid. The formation of tar is practically negligible and only 1% of mercury (on the acetic acid) is required as against 4% by known processes. (B) In carrying out the process described under (A) the mercuric salt may be replaced by metallic mercury in the presence of ferric sulphate. At the end of the reaction the ethylidene diacetate, carrying the ferrous sulphate formed, may be drawn off and the mercury left in the apparatus, a feature which makes a continuous process possible. From 50 pts. of metallic mercury, 1000 pts. of glacial acetic acid, 20 pts. of anhydrous ferric sulphate, 65 pts. of acetic anhydride, and a mixture of 50 pts. of sulphoacetic acid and 125 pts. of glacial acetic acid, 1500 pts. of crude, or 1200 pts. of purified ethylidene diacetate are obtained.
R. BRIGHTMAN.

Manufacture of *n*-butyl esters of aminobenzoic acids. E. C. R. MARKS. From ABBOTT LABORATORIES (E.P. 252,870, 12.5.25).—*n*-Butyl esters of aminobenzoic acids are obtained by the action of 2 mols. of *n*-butyl halides (preferably *n*-butyl bromide) on a metal salt of the aminobenzoic acid in the presence of a catalyst, such as diethylamine, dipropylamine, dibutylamine, etc., or copper powder. Alternatively the metal salt of a nitrobenzoic acid may be used and the *n*-butyl nitrobenzoate reduced by means of iron and hydrochloric acid. *n*-Butyl *p*-nitrobenzoate has b.p. 160°/6 mm., m.p. 33—35°; *n*-butyl *p*-aminobenzoate, m.p. 57—58·5°.
R. BRIGHTMAN.

Production of pure lecithin. C. H. BOEHRINGER SOHN, Assees. of O. DENGLER (U.S.P. 1,586,145, 25.5.26. Appl., 11.5.25).—By treating 100 g. of sodium cholate dissolved in 100 c.c. of water with 20 g. of lecithin, a soluble double compound of lecithin and the alkali cholate is formed. It is precipitated by addition of alcohol and ether and is completely freed from fatty impurities and cholesterol by treatment with ether or benzene. The purified product is dissolved in water and decomposed into lecithin and cholic acid by treatment with 10% aqueous hydrochloric acid and the purified lecithin is recovered by extraction with ether. Alternatively an aqueous solution containing lecithin and sodium cholate is extracted with ether and evaporated to dryness under reduced pressure, when a portion of the lecithin can be directly recovered from the residue by extraction with ether. The remainder is recovered, after treatment of the extracted residue with dilute hydrochloric acid, by a further extraction with ether.
T. S. WHEELER.

Apparatus for generating ether. F. E. LICHTENTHAELER (U.S.P. 1,587,161, 1.6.26. Appl., 28.9.23).—A reservoir for sulphuric acid is provided with a series of tubes of acid-resisting material (lead) connected to it at the top and bottom. Each of the tubes is surrounded by a steam jacket and circulation of the acid in the reservoir through the tubes is thus induced. Alcohol is introduced into each of the tubes by means of a perforated pipe extending some distance into it.
T. S. WHEELER.

Manufacture of a pharmaceutical product. [Compound of diallylbarbituric acid and 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone.] A. THIELE, Assr. to CHEMISCHE FABRIK AUF ACTIEN (VORM. E. SCHERING). (U.S.P. 1,588,554, 15.6.26. Appl., 18.5.23).—See E.P. 198,379; B., 1924, 114.

Antipyretic. [*o*-Acetoxy-*p*-methoxybenzoic acid.] W. SCHULEMANN and F. SCHÖNHÖFER, Assrs. to WINTHROP CHEMICAL CO., INC. (U.S.P. 1,588,814, 15.6.26. Appl., 21.11.25. Conv., 4.4.24).—See E.P. 231,886; B., 1925, 942.

Process of making olefine oxides. Process of making propylene oxides. J. N. BURDICK, Assr. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,589,358–9, 22.6.26. Appl., 7.8.23 and 14.11.25).—See E.P. 236,379; B., 1925, 738.

Manufacture of ethylsulphuric acid. A. A. L. J. DAMIENS, M. C. J. E. DE LOISY, and O. J. G. PIETTE (U.S.P. 1,589,372, 22.6.26. Appl., 6.12.19. Conv., 5.9.19).—See E.P. 152,495; B., 1920, 833 A.

Manufacturing acetic anhydride. L. HÖRMANN and F. KAUFLER, Assrs. to A. WACKER-GES. FÜR ELEKTROCHEM. IND. G.m.b.H. (U.S.P. 1,590,097, 22.6.26. Appl., 2.2.24. Conv., 12.2.23).—See E.P. 211,167; B., 1924, 890.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effect of evacuation of a photographic plate on its sensitivity. O. MASAKI (Mem. Coll. Sci. Kyoto, 1926, A 9, 285—302).—Various commercial plates were exposed to light from a Mazda lamp, using a Hurter and Driffield wheel to give differences of exposure, and the density of the plates was measured by means of a photo-electric cell. In all cases the plate which had been subjected to reduced pressure showed greater density than the corresponding untreated plate, though to a greatly varying extent; for Ilford panchromatic plates the effect is small. For many plates the contrast is increased, but for Ilford panchromatic plates the opposite effect is produced. The lower the pressure the greater is the effect produced. In the case of red light, however, the opposite effect was observed, the treated plate being less sensitive than the untreated one; on the other hand, the contrast is increased; thus, treatment reduces fog. For dyed plates the sensitising action of the dye is generally enhanced. Experiments on drying the plates indicated that the main effect of evacuating the plates was the removal of moisture and the change in sensitivity and contrast was due to the dryness of the emulsion. E. B. LUDLAM.

Photographic reversal. H. BELLIOT (Compt. rend., 1926, **182**, 1609—1612).—An investigation of the photographic properties of red and infra-red radiation. A previously fogged plate, on which reversal has been produced by radiation of wave-length greater than $0·7\mu$, recovers much of its sensitivity to visible light, whilst the same plate, solarised, is no longer sensitive. Radiation which produces reversal on a fogged plate blackens unused plates. If the portion of the spectrum between 0·5 and $0·95\mu$ falls on a plate fogged in one portion and solarised in another the radiations of greater wave-length reverse the fogged portion and blacken the solarised portion. The radiations of shorter wave-length, however, enhance the existing solarisation.
J. S. CARTER.

Spectral distribution of sensitivity of photographic materials. JONES and SANDVIK.—See A., July, 694.

PATENTS.

Production of photographs on parchment, paper, etc. E. E. JELLEY (E.P. 253,380, 24.7.25).—Any pure paper is sensitised by coating with a solution containing a soluble silver salt, in which the silver is combined with one or more of the three higher halogens, and a soluble, non-volatile, crystallisable weak acid, *e.g.*, citric acid, tartaric acid, etc. The best silver salt is silver chlorate, but silver perchlorate, silver bromate, and silver salts of organic acids containing substituted halogens, soluble in water to a greater extent than 0·01 pt. in 100 pts. by weight, may be used. It is preferable to coat with the silver salt first, dry, and then coat with an alcoholic solution of the acid. The paper is exposed to give a faintly visible image and is developed in a solution of an organic reducer such as metol, quinol, metol-quinol, or pyrogallol. By suitable choice of exposure time and developer, a range of tones from warm brown to black is obtained. After development the paper is well washed and fixed in a solution containing sodium thiosulphate and sodium bicarbonate, or in an acid fixing bath. If the paper is sensitised to the extent of less than 0·0005 g. of silver salt per cm.^{2}, a soluble silver salt is added to the developer and shorter exposure times are used. The hydrogen-ion concentration of the developer should be kept within the limits 10^{-4} and 10^{-2}. Amidol tends to fog the paper. W. CLARK.

Photographic developer. K. BINDER (E.P. 253,732, 14.7.25).—See F.P. 600,801; B., 1926, 566.

XXII.—EXPLOSIVES; MATCHES.

Method for the determination of the velocity of detonation over short lengths of explosive. E. JONES (Safety in Mines Res. Bd., Paper 22, 1926, 21 pp.).—The apparatus is arranged so that a travelling detonation wave is caused to break two electrical circuits at known distances (*e.g.*, 2 cm.) from each other. The first break causes a charged condenser of known capacity to discharge through a known resistance; the second break causes the discharge to cease. Measurement of the residual charge in the condenser enables the time of discharge to be calculated. The velocity of detonation obtained by this method was somewhat less than 3% lower than that obtained over 1 m. of the same TNT fuse by the Mettegang method (5th. Int. Congr. Appl. Chem., Berlin, 1903, **2**, 322). S. K. TWEEDY.

PATENTS.

Gelatinising nitrocellulose in nitro-compound powders and celluloid. L. PARODI-DELFINO (E.P. 252,978, 28.10.25).—Phthalide or its homologues are claimed as gelatinisers and stabilisers in the manufacture of celluloid and of nitrocellulose powders which may or may not contain nitroglycerin. These compounds have the advantage of being non-volatile and enable cool-burning colloided nitrocellulose powders containing a reduced proportion of nitroglycerin to be obtained without the use of volatile solvents. A suitable composition comprises nitrocellulose 12 pts., nitroglycerin 4—6 pts., and phthalide or its homologues 1—4 pts.
T. S. WHEELER.

Primer composition. H. RATHSBURG (U.S.P. 1,586,380, 25.5.26. Appl., 18.3.25. Conv., 23.10.23).—The composition contains guanylnitrosoaminoguanyltetracene. L. A. COLES.

XXIII.—SANITATION; WATER PURIFICATION.

Effect of chlorination on trickling sewage filters. M. M. COHN (Eng. News-Rec., 1926, **96**, 943—948).—At Schenectady, N.Y., the continuous application of liquid chlorine in quantities equivalent to 4—30 p.p.m., to the Imhoff tank effluent, prior to filtration, did not materially affect the nitrification efficiency of healthy filters, although the tank

effluent itself was rendered sterile by the treatment. Odours were reduced proportionately to the quantity of chlorine applied. Biological growths present in the nozzles of sprays and in the distribution pipes were destroyed and removed, the nozzles subsequently remaining in a clean condition. Another effect of the chlorination was the removal of the gelatinous films from the surface of the beds; in consequence, "ponding" did not occur and nuisance from flies was substantially reduced. Bleaching powder gave results similar in character, and is a suitable reagent to use for fly control. Dichlorobenzene mixture when sprayed on the walls and sheltered spots about a filter rapidly destroys adult *Psychoda*, mosquitos, and young spiders.

W. T. LOCKETT.

Determination of p_H in natural waters. I. R. NASINI and C. PORLEZZA (Annali Chim. Appl., 1926, **16**, 156—166).—The results obtained in the electrometric and colorimetric determination of the p_H value of natural waters are discussed and the special sources of error and means of avoiding them indicated. Determination of this magnitude is of particular value in following the variation in the proportion of carbon dioxide dissolved in these waters, and in tracing the alteration of sulphur waters, especially as regards the oxidation of the hydrogen sulphide.

T. H. POPE.

Prevention of corrosion and "red water." J. R. BAYLIS (J. Amer. Water Works Assoc., 1926, **15**, 598—633).—The fundamental factors influencing corrosion rates of iron, zinc, lead, and cement-lined pipes are discussed and experimental results relating thereto are given. The problem of prevention of corrosion in water mains is one of protection by film formation. Films may be formed by precipitating the products of corrosion on the metal surface or by producing a supersaturation of some compound in the water, *e.g.*, calcium carbonate, which will form an impervious coating. No ordinary compound of iron will exist in solution in most waters in quantities greater than 0·1 p.p.m. of soluble iron when the water is at calcium carbonate equilibrium or more alkaline, within the range of natural waters. When the water is at this point or more alkaline the rate of solution of iron is comparatively slow and all products of corrosion where dissolved oxygen is present are quickly precipitated. The amount of calcium carbonate which will be held in solution depends upon the alkalinity, hydrogen-ion concentration, and to a slight extent upon the amount of neutral salt present. When it is not desired to produce a coating of calcium carbonate on the pipe surface the p_H of the water should be so adjusted that very little soluble iron will exist in the water in the absence of dissolved oxygen and at the same time calcium carbonate supersaturation will be avoided. Values of p_H suitable for this purpose for waters with varying alkalinities are plotted. These vary from p_H 8·5 with water having an alkalinity of 12, to 8·25, 7·8, 7·5, 7·4, and 6·75 with alkalinities of 25, 50, 75, 100. and 350 respectively. Treatment along these lines prevents staining and reduces "red water" to an occasional occurrence. When it is desired to form a protective coating of calcium carbonate, lime should be added to raise the p_H value to 9·5 with an alkalinity of 25, or to 8·5, 8·0, 7·8, 6·9 with alkalinities of 50, 75, 100, 350, respectively. After a coating has been formed the water may be treated so as to obtain p_H values intermediate between the two series given, at which values, with the respective alkalinities, the calcium carbonate is substantially in equilibrium.

W. T. LOCKETT.

***Coli-aerogenes* group [of bacteria] in soil.** S. A. KOSER (J. Amer. Water Works Assoc., 1926, **15**, 641—646).—Members of the *coli-aerogenes* group were found to be fairly abundant in the soils of cultivated fields and pastures. The results of the methyl-red, Voges–Proskauer, and citrate tests brought out a general correlation between the proportion of the various sections of the colon group and the apparent degree of fæcal pollution of the soil. The proportion of the *Bact. coli* type (MR+, VP—, citrate—) increased with the amount of fæcal contamination, being highest in the soil from pastures.

W. T. LOCKETT.

Fluosilicates [silicofluorides] as insecticides. S. MARCOVITCH (Ind. Eng. Chem., 1926, 18, 572—573). —Sodium silicofluoride consisting of 70—75% of the salt and the remainder alumina and occupying a volume of 60 cub. in. per lb., and calcium silicofluoride, a by-product in the volatilisation method of treating phosphate rock for phosphoric acid, with a volume-weight of about 70 cub. in. per lb. in one form and 120 cub. in. in the other, were used in field trials and found effective against a variety of insects not controlled by arsenic. These substances are also far less toxic to man than arsenicals. Flea beetles, and blister and striped cucumber beetles were readily controlled, and the bacterial spot disease of the peach was successfully combated with sodium silicofluoride at dilutions of 1 to 4000 or 4500.

D. G. HEWER.

Danger of mercury vapour. STOCK.—See A., July, 707.

PATENTS.

Compound having bactericidal and fungicidal properties [from sulphite-cellulose waste lye]. E. SCHLUMBERGER (U.S.P. 1,585,792, 25.5.26. Appl., 26.10.23).—1000 kg. of concentrated sulphite-cellulose waste liquor are diluted with 500 litres of water and treated with chlorine. The separated yellowish-red mass is filtered off and dried. It contains 25—28% Cl, is soluble in water, and possesses strong bactericidal and fungicidal properties. The properties of the disinfectant are improved by a subsequent treatment with an oxidising agent such as potassium chlorate and hydrochloric acid.

E. S. KREIS.

Centrifugal liquid purifiers. (E.P. 252,421). —See I.

Electrometric determination of acidity or alkalinity of water (G.P. 413,043).—See XVI.

Fungicide (U.S.P. 1,587,977).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPTEMBER 3, 1926.

I.—GENERAL; PLANT; MACHINERY.

Protecting thermocouples by transparent silica tubes. R. A. Ragatz and O. A. Hougen (Chem. Met. Eng., 1926, **33**, 415).—A thermocouple enclosed in an ordinary translucent fused silica protection tube when used at above 900° in a reducing atmosphere shows a falling off in E.M.F. due to the porosity of the tube under these conditions. This was found not to be the case with transparent silica tubes tested up to 1200°. Devitrification of the tube is also very much less than with the older type of fused silica. C. Irwin.

See also A., Aug., 787, **Temperature of vapour evolved from solutions** (Reissmann). 789, **Adsorption from viscous media by charcoal** (Weissenberger, Baumgarten, and Henke). 800, **Heats of adsorption and problem of promoter action** (Fryling).

Patents.

Reverberatory furnaces. B. Versen (E.P. 243,751, 26.11.25. Conv., 27.11.24).—A regenerative reverberatory furnace is provided with ports for combustible gas and air situated behind each other on the centre line of the furnace, the two gases arriving in an upward direction. Converging streams of a pressure medium (compressed air) are directed across the gas ports and are inclined downwards so that the most intense heat is generated on the surface of the bath in the centre of the furnace and the walls and roof are at a relatively lower temperature. The jets of pressure medium may vary in size and velocity to improve the mixing, and the nozzles are protected from the heat by a fireproof casing arranged in a recess in the furnace end, also, if desired, by water cooling; and a slight flow of pressure medium is allowed to pass through the fireproof casing even when the operation of the furnace is reversed. B. M. Venables.

Pulverising or grinding mills. B. Scherbaum (E.P. 245,435, 15.12.25. Conv., 31.12.24).—The fixed portion of the apparatus consists of a vertical cylinder provided on its inner surface with teeth arranged in courses; the number of teeth in a course increases, but their pitch and thickness decrease from the top downwards, *i.e.*, in the general direction of the material being ground. The moving portion of the apparatus comprises a vertical shaft having outwardly projecting teeth in courses intercalating with the courses of the fixed part. Both the moving and fixed portions are divided horizontally into sections so that the parts may be readily changed for others more suitable to the material being ground, and the fixed portion is made slightly expansible (by dividing it segmentally) so that the clearances may be altered. B. M. Venables.

Grinding or crushing machines. W. J. Booth (E.P. 253,817, 29.12.25).—The grinding is effected between a fixed outer annular ring of abrasive material and two or more curved segments which are dragged around the interior surface of the ring by drag-links, centrifugal force being sufficient to keep them in contact. B. M. Venables.

Pulverising mills. E. C. Loesche (E.P. 247,562, 10.2.26. Conv., 12.2.25).—The rotating grinding track of a pulveriser is combined in one piece with the extraction fan. B. M. Venables.

Pulverising mill. L. C. Bonnot and W. M. Barker, Assrs. to Bonnot Co. (U.S.P. 1,589,740—1, 22.6.26. Appl., [a] 30.7.25, [b] 4.11.25).—(a) An exhaust fan communicates directly with the rotary drum of a ball mill, and means are provided for positively preventing the grinding balls from entering the fan. (b) A rotary drum containing grinding balls is provided at one end with a feed opening and at the other end with an axial discharge outlet in the form of a cone flared inwards. The larger end of the cone is unobstructed. Means are provided for impelling a current of air from the inlet to the outlet and for repelling the grinding balls and coarse material from the mouth of the discharge cone. H. Holmes.

Centrifugal machines. A. G. Enock (E.P. 253,653, 14.4.25).—The liquid to be treated is fed to the interior of an inner rotating bowl, and passes through perforations in the back of the bowl to an outer bowl rotating at the same speed, and is finally discharged through other perforations at the front end in the extended wall of the inner bowl; the two portions of the inner bowl are divided by an annular baffle through which the feed pipe is introduced. Separated solid matter is collected in pockets formed by vanes in the space between the bowls, the vanes being made of spiral form to guide the liquid through the space from back to front. B. M. Venables.

Apparatus [horizontal centrifuge] for continuously separating liquids from solids. H. C. Behr (U.S.P. 1,589,097, 15.6.26. Appl., 4.12.23).—In a continuous horizontal centrifuge for separating liquor from granular aggregates, spaced inner and outer concentric foraminous cones are rotated at different speeds about a horizontal axis. The inner cone carries a screw conveyor for moving

the material over the inner surface of the outer cone from the small to the large end. The outer cone carries, integral with it, means for gathering and expelling outwards in a narrow zone undiluted liquor separated at the small end of the cone, and other means for gathering and expelling outwards, in a narrow zone spaced from the first zone, diluted liquor separated at the large end of the cone. A movable partition is provided for adjusting the proportion of diluted to undiluted liquor, and receptacles are arranged to receive these liquors and the washed solids discharged over the large end of the outer cone.
H. Holmes.

Evaporator. M. J. Kermer (U.S.P. 1,588,029, 8.6.26. Appl., 20.3.24).—The liquid to be evaporated or dehydrated is fed to the interior of a rotating vessel, which is provided with a rotating steam jacket. The stream of liquid passes down the interior of the vessel and is discharged at the bottom. The centrifugal force causes water from the condensed heating steam to be thrown outwards, with the result that the heating efficiency is unusually good, since there is no film of water on the heat-transmitting surface.
E. S. Kreis.

Apparatus for dehydrating liquids. L. A. Zohe (U.S.P. 1,588,929, 15.6.26. Appl., 28.2.21).—A centrifugal fan mounted in a closed chamber is provided with an inlet for the liquid and for a moisture-absorbing gas. The mixture is thrown from the fan against a surrounding wall rotating about the axis of the fan, so that the liquid forms a film on the wall. H. Holmes.

Filtering material. W. H. Hoodless (U.S.P. 1,589,531, 22.6.26. Appl., 10.3.21).—Kieselguhr having its internal structure lined throughout with a thin film of finely-divided decolorising carbon is prepared from an intimate mixture of dry kieselguhr and dry sawdust, which is placed in a retort closed except for a small opening. The retort is heated until a combustible vapour escapes from the opening, and the temperature is then maintained constant until combustible vapour ceases to escape.
H. Holmes.

Decolorising and filtering material. W. H. Hoodless (U.S.P. 1,589,532, 22.6.26. Appl., 10.3.21; cf. preceding abstract).—The internal structure of particles of kieselguhr is lined with a thin film of carbon from heat-charred molasses.
H. Holmes.

Filter-press. F. J. Bisbee (U.S.P. 1,589,834, 22.6.26. Appl., 12.1.25).—Filter-plates are mounted face to face on an unobstructed longitudinal support. Stationary members, one at each end of the support, serve alternately as abutments toward which the plates are moved when they are to be separated. A movable head is mounted near each end of the support, and each head is provided with means by which it is actuated to move the plates toward the stationary head at the other end. H. Holmes.

Retort. S. Forssén (U.S.P. 1,589,746, 22.6.26. Appl., 25.11.24. Conv., 21.12.23).—The material to be treated is placed in an inner vessel having its walls spaced concentrically from an outer casing. The bottom of the vessel is frusto-conical with a central aperture, and the top is provided with an aperture in axial alinement with the bottom aperture. A cover on the casing is spaced from the top of the vessel, and the bottom of the casing is spaced from the bottom of the vessel. These spaces communicate freely with the annular space around the vessel, and a draught-inducing device is mounted in the aperture at the bottom of the vessel for circulating air, heated in the annular space, through the vessel.
H. Holmes.

Apparatus for subjecting gases or vapours to the absorptive action of solids. Farbw. vorm. Meister, Lucius, & Brüning (E.P. 239,189, 11.8.25. Conv., 28.8.24).—See G.P. 421,027; B., 1926, 425.

Apparatus for intimately mixing gases and liquids. Farbenfabr. vorm. F. Bayer & Co., Assees. of E. Stroder (U.S.P. 1,592,231, 13.7.26. Appl., 8.6.23. Conv., 20.6.22).—See E.P. 199,718; B., 1924, 81.

Rotary drying machine. J. A. Reavell (U.S.P. 1,591,053, 6.7.26. Appl., 12.11.23. Conv., 28.2.23).—See E.P. 216,577; B., 1924, 621.

Pulverising machine. E. Charton and P. M. Mongereau (U.S.P. 1,591,283, 6.7.26. Appl., 26.11.23. Conv., 25.10.23).—See E.P. 223,582; B., 1925, 655.

Machine for pulverising or grinding coal and other materials. P. W. Griffin, Assr. to Vickers, Ltd. (U.S.P. 1,591,758, 6.7.26. Appl., 20.7.23. Conv., 25.7.22).—See E.P. 205,873; B., 1924, 7.

Extraction, solution, and mixture of soluble and insoluble substances. N. Bendixen, Assr. to F. R. M. Co., Ltd. (U.S.P. 1,592,713, 13.7.26. Appl., 20.4.25. Conv., 27.8.24).—See E.P. 242,020; B., 1926, 2.

Heat exchangers. G. K. Engelhart, Assee. of C. B. Grady (E.P. 242,231, 30.6.25. Conv., 30.10.24).

Absorption refrigerating apparatus. Mannesmann Kälte-Ind. A.-G. (E.P. 244,725, 6.11.25. Conv., 17.12.24).

Liquid fuel furnaces. A. Laing (E.P. 254,618, 3.2.26).

Surface condensers. Mirrlees Watson Co., Ltd., and W. A. Dexter (E.P. 254,626, 13.2.26).

Extraction process and apparatus (U.S.P. 1,587,646).—See XVII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Constitution of coal. S. W. PARR (Ind. Eng. Chem., 1926, **18**, 640—648).—On submitting normal coal to a current of oxygen at 100° a steady increase in weight occurs, with the formation of moisture and carbon dioxide. In 45 days 8·5% of moisture was formed and 6% of carbon dioxide, and the coal increased in weight about 7%. At normal temperature 6% of moisture and 1% of carbon dioxide only were formed in 46 weeks, the increase in weight being 2%. Very different results are obtained by heating unoxidised and oxidised lignitic and bituminous substances (the portions of coal insoluble and soluble respectively in phenol) to about 300°. Thus 100 g. of fresh lignitic substance evolved 153·2 c.c. of carbon dioxide, but the oxidised substance evolved 1423·0 c.c. Fresh bituminous material evolved 462·5 c.c., but oxidised bituminous material evolved only 244·0 c.c. When oxidised lignitic substance (insoluble in xylene) and fresh bituminous substance (soluble in xylene) were re-mixed, it was found that the coking property of the mixture had been destroyed by the oxidation of the lignitic substance. In experiments to determine the melting or softening point of coal it was found that this was associated with the bituminous substance alone; the melting or coking property is destroyed by oxidation. Experiments on coal carbonisation at about 750° are described. H. MOORE.

Adsorption of carbon dioxide by coal. E. SINKINSON and H. G. TURNER (Ind. Eng. Chem., 1926, **18**, 602—605).—To study the adsorption of carbon dioxide a 218-c.c. container was used closed by a stopper at the bottom. This was fitted with an axial thermocouple extending to the inside of a cup introduced from the bottom of the container and containing about 10 g. of the coal or charcoal to be tested. Means were provided for exhausting the flask and for introducing a measured volume of carbon dioxide, which remained in contact with the coal for 45 min. The adsorbed gas is held very tenaciously, the last traces being removable with difficulty under a vacuum of 0·003 mm. High-grade coals are more adsorptive than low-grade coals, and the same remark applies to the charcoals (fixed carbon residues) from such coals. Moist carbon dioxide is adsorbed to a greater extent than the dry gas. In the adsorption of oxygen by coal the volume of carbon dioxide recovered is not a true index of the amount of oxidation taking place. The adsorption of carbon dioxide from mixtures of carbon dioxide and oxygen is proportional to its partial pressure. The rise in temperature is generally proportional to the volume of gas adsorbed. The adsorption of carbon dioxide produces a greater rise in temperature than the adsorption of oxygen. H. MOORE.

Friability tests on various fuels sold in Canada. J. H. H. NICOLLS (Dept. of Mines, Canada, Investigation of Fuels and Fuel Testing, 1924, [644], 20—35).—Three sizes of fuel (stove size, $1\frac{1}{2}$ in.—2 in.; nut size, 1 in.—$1\frac{1}{2}$ in.; pea size, $\frac{1}{2}$ in.—1 in.) were examined by means of shatter and tumbling tests. The following order in friability of fuels was obtained: peat the least friable; by-product coke, Pennsylvania anthracite, Welsh anthracite, bituminous coals and lignites; and semi-bituminous or "smokeless" coals. Weathering under cover increased the friability of the lignites; weathering in the open increased considerably the friability of all the fuels except the coke, but even after weathering, the peat was more resistant to breakage than most of the unweathered fuels. A. C. MONKHOUSE.

Effects of exposing Canadian lignite to atmospheres of different humidities. J. H. H. NICOLLS (Dept. of Mines, Canada, Investigation of Fuels and Fuel Testing, 1924, [644], 36—44).—Typical Alberta lignites were air-dried under different conditions of humidity comparable with summer and winter humidities and also at a standard relative humidity of 60%, using calcium chloride solution of *d* 1·30. Using similar conditions of humidity the minimum percentage of moisture to which a lignite will dry is higher than the maximum value obtained when starting with the same lignite dried at 90°. For the purposes of classification of the lignites air-drying must be carried out under standard conditions of humidity. A. C. MONKHOUSE.

Coking experiments on coals from the Maritime Provinces. B. F. HAANEL and R. E. GILMORE (Dept. of Mines, Canada, Investigation of Fuels and Fuel Testing, 1924, [644], 1—19).—An investigation of the suitability of the coking coals of New Brunswick and Nova Scotia for the production of domestic coke is described. Laboratory tests and large-scale tests in a battery of Semet-Solvay coke ovens were made together with tests on the combustibility of the resulting cokes. 52 Nova Scotia coals had average ash and sulphur contents of 11% and 3·3% respectively as compared with 24 New Brunswick coals with 15% and 6·9%. Satisfactory cokes for domestic purposes were obtained by blending, and washing the coals reduced the ash and sulphur contents. A. C. MONKHOUSE.

Report of carbonisation and washing experiments on sub-bituminous coal from Coal Valley, Alberta. R. A. STRONG (Dept. of Mines, Canada, Investigation of Fuels and Fuel Testing, 1924, [644], 60—68).—Coal mined from the open quarry section of the mine gave on analysis, lump: ash 8·7%, calorific value 11,255 B.Th.U. per lb.; slack: ash 21·2%, calorific value 9325 B.Th.U. per lb. By washing, the ash of the "slack" coal was reduced to 15%. A laboratory carbonisation at 600° yielded $8\frac{1}{2}$ gals. of tar per ton and the calorific value of the coke produced was 15% higher than that of the original coal. The fuel is unsuitable for domestic fuel unless briquetted. A. C. MONKHOUSE.

Distillation of oil shale. A. A. SWINNERTON (Dept. of Mines, Canada, Investigation of Fuels and Fuel Testing, 1924, [644], 69—81).—The different methods used by the Canadian Dept. of Mines and U.S. Bureau of Mines for the evaluation of oil shales

are compared (cf. Gilmore and Swinnerton, B., 1926, 116). With a particular shale the yields of oil in gals. per short ton were 28 with the horizontal tube, 29 with the Scotch tube, 30 with the cross retort in a lead bath, 31 with the large field-assay retort, and 32 with the small field-assay retort. Excessive cracking of the heavier oils occurred with the horizontal tube retort. The use of steam, particularly in the small field-assay retort (350-g. charge), did not materially affect the yield of crude oil but caused a decrease in the light oils and an increase in the heavy oils. A. C. MONKHOUSE.

Hydrogen from water-gas. R. M. EVANS and W. L. NEWTON (Ind. Eng. Chem., 1926, 18, 513—517).—The conversion of a mixture of water-gas and steam into hydrogen and carbon dioxide may be effected catalytically by passing the mixture over metal oxides. At temperatures of 380° and 444° cobalt oxide and iron oxide are the most active single-component catalysts, and the most generally suitable catalyst is composed of precipitated cobalt (or iron) oxide with a small admixture of aluminium oxide (2—5%) and potassium oxide (1%). The cobalt catalyst is gradually and permanently poisoned by traces of hydrogen sulphide, carbon disulphide, and carbon oxysulphide. The iron catalyst is rendered less active by traces of hydrogen sulphide and carbon oxysulphide, but regains its original activity when the pure water-gas mixture is again passed over it. The poisoning action of a small concentration of carbon disulphide on the iron catalyst is similar to, but about ten times as powerful as, that of hydrogen sulphide and carbon oxysulphide. W. T. K. BRAUNHOLTZ.

Reduction of carbon monoxide under ordinary pressure. A. JAEGER and H. WINKELMANN (Abhandl. Kennt. Kohle, 1925, 7, 55—62; Chem. Zentr., 1926, I, 3517—3518).—The yields of methane and of mixed oxides of carbon obtained by passing a mixture of 26% of carbon monoxide and 67% of hydrogen over a nickel–pumice catalyst are, respectively, 12% and 12% at 220°, 54% and 7% at 245°, and 66% and 6% at 250°. The optimum temperature for methane formation lies in a narrow zone at 250°. A mixed copper–nickel catalyst yielded only 6% of methane; no ethylene could be detected when a palladium catalyst was used. Contrary to the results of Church (F.P. 519,649) no formaldehyde is formed when a mixture of carbon monoxide, hydrogen chloride, and hydrogen is passed over cuprous chloride, copper, iron, or nickel at 200—300°; it is produced in considerable amounts, however, when formic acid vapour mixed with hydrogen is passed over metals at lower temperatures, or over indifferent substances at higher temperatures or through an empty glass tube at 700—750°. Carbon monoxide and steam yield small amounts of formic acid when passed over hydrated silica at 320°; at 450° in a quartz tube about 1·5% of the carbon monoxide is converted into formaldehyde. This yield decreases with rising temperature and increasing streaming velocities (100 c.c./min.) and no formaldehyde is formed at all when copper– or nickel–pumice catalysts are used or when the steam is replaced by hydrogen. Hydrogen free from hydrogen sulphide or prepared by dehydrogenation of tetralin (tetrahydronaphthalene) does not reduce carbon monoxide to formaldehyde at 400°. Formaldehyde likewise is not obtained by reducing the compound $K_3Fe(CN)_5,CO$ with hydrogen. S. K. TWEEDY.

Coal gas poisoning. Resuscitation experiments on animals with lobeline. DOLLINGER (Gas- u. Wasserf., 1926, 69, 561—566).—Experiments on apes show that symptoms of poisoning from illuminating gas, due to the presence of carbon monoxide, appear almost as soon with low concentrations of carbon monoxide as with high. Injection of lobeline effects resuscitation much more rapidly than artificial respiration. Cardiac injection gave the quickest results but the results of intramuscular injection also were mostly good. Unfavourable after-effects were not observed, and it is concluded that injection of lobeline affords a rapid means of resuscitation in cases of carbon monoxide poisoning. R. BRIGHTMAN.

Cresylic acid from petroleum distillates. L. J. CATLIN (Ind. Eng. Chem., 1926, 18, 743—744). —Cracked petroleum distillates on washing with caustic soda yielded 0·007% of their volume of phenols. The oil was precipitated from the soda solution by saturation with hydrogen sulphide from still gases. The aqueous portion was brought to the composition corresponding to the normal sulphide by addition of further caustic soda and concentrated to b.p. 143°, when on cooling a further separation of oil took place and the residue could be converted into commercial 62% sodium sulphide. The phenol fraction distilled mainly between 195° and 227°; it gave tests for cresylic acid (Allen, "Commercial Org. Anal.," 4th ed., 3, 316) but contained no cresols, having *d* 0·995 to 1·013. C. IRWIN.

See also A., Aug., 805, **Extinction of methane flames by diluent gases** (COWARD and HARTWELL). 807, **Catalytic oxidation of carbon monoxide** (HOSKINS and BRAY).

Determination of acetone. MARASCO.—See XX.

PATENTS.

Coal washing. A. ROBINSON, and SIMON-CARVES, LTD. (E.P. 252,475, 5.3.25).—The coal, prior to washing, falls on an inclined sieve which removes a portion of the fine coal. The sieve is of wedge shape ⅛in. mesh, fitted with springs, and is placed in the shoot to the washer. The fine coal is mixed with the drained slurry from the washer and the product is of suitable moisture content to be used as a fuel. A. C. MONKHOUSE.

Briquetting. W. TYRRELL (U.S.P. 1,588,823, 15.6.26. Appl., 31.12.24).—A mixture of equal parts of caustic soda and diatomaceous earth is moistened with water and heated at 105° for ½ hr. Two per cent. of this mass is then mixed with the

finely ground material to be briquetted and the mass compressed. S. Pexton.

Operating vertical chamber ovens to produce water-gas. N. V. Silica en Ovenbouw Mij., Assees. of C. Otto & Co. (E.P. 240,468, 23.9.25. Conv., 25.9.24).—Vertical retorts or chamber ovens are provided with two coke-discharging doors each of which discharges coke from the half of the retort immediately above it. Coke is discharged intermittently and alternately from the two halves of the retort. In this way the coke mass within the retort is broken up and steaming of the retorts is more effective. S. Pexton.

Preheating of fuel preliminary to carbonisation. J. Rude (E.P. 252,988, 12.11.25).—The fuel entering a vertical retort is preheated in a chamber above the retort by circulating a portion of the waste gases from the combustion flues by a fan or injector through the chamber. The sensible heat of the coke may be used in place of the waste gases. An alternative method is to combine with the above a heat exchanger which is heated by the flue gases or by gases heated by the coke. The preheated fuel is fed into the retort through shoots which act as seals and are also jacketed by the waste gases. A. C. Monkhouse.

Gas-retort settings. H. J. Toogood, and R. Dempster & Sons (E.P. 253,676, 8.5.25).—In order to economise space and plant fittings retorts at the same height are connected in pairs to the same ascension pipe, there being ascension pipes at both ends of the retorts. An easily operated baffle or butterfly valve is provided to close the offtake from each retort, when discharging and charging the retort. S. Pexton.

Manufacture of gas. Woodall-Duckham (1920), Ltd., E. W. Smith, and T. C. Finlayson (E.P. 253,702, 26.5.25).—In order to increase the efficiencies of water-gas manufacture and tar-cracking in vertical retorts it is proposed to divide the process into two stages. The minimum amount of steam to quench the coke is introduced into a number of the retorts, the tops of which are maintained at a lower temperature than usual. The remaining retorts are fed with broken coke and a liberal supply of steam is admitted. Water-gas is more efficiently made because the coke charge is not consolidated. Tar is sprayed on to the coke at a point where the temperature is suitable for cracking. S. Pexton.

Apparatus for producing solid [carbonised] fuel. M. J. Trumble (U.S.P. 1,586,131, 25.5.26. Appl., 9.2.24).—A retort has an opening for the introduction of carbonaceous material, which opening may be closed gas-tight. An eduction pipe extends through one end wall of the retort and has an open end adjacent to the other end of the retort. There are also means for heating the retort, and a plunger to force material from the retort into the open end of the eduction pipe. H. Moore.

Coke-ovens. A. Roberts, Assr. to Chicago Trust Co. (U.S.P. 1,588,469, 15.6.26. Appl.,20.11.21. Renewed 5.2.26).—The ovens are heated by vertical flues each divided into an upper and lower chamber. Every chamber is provided with two air ports and one gas nozzle, the air ports being connected to separate regenerators which can be independently controlled. S. Pexton.

Production of highly-active charcoal from wood. P. Wöhler, Assr. to Rhenania Ver. Chem. Fabr. A.-G. (U.S.P. 1,588,868, 15.6.26. Appl., 1.9.21. Conv., 14.4.17).—Small pieces of wood are carbonised at 300—500° with the exclusion of air. The product is incorporated with alkali and heated at bright redness with the exclusion of air. S. Pexton.

[Carbonisation] retort. F. Pavelik (U.S.P. 1,589,217, 15.6.26. Appl., 22.12.22).—The retort consists of a rectangular casing within which a number of horizontal parallel conveyors operate separately over mechanically driven toothed rollers at opposite ends of the retort. Fuel fed into the retort drops on to the uppermost conveyor, which feeds it on to the next lower one. In this way a shallow layer of fuel can be carbonised at varying rates. S. Pexton.

Apparatus for extracting volatile oils from solid substances. McG. Cline (U.S.P. 1,589,838, 22.6.26. Appl., 24.8.23).—The solid substance from which the volatile oil is to be distilled is admitted at the top of a retort and passes down over a series of stationary plates, being transferred by scrapers attached to a rotating shell surrounding the fixed plates. The spent material is discharged at the bottom of the retort. S. S. Woolf.

Gas purification. W. H. Fulweiler, Assr. to U.S.I. Contracting Co. (U.S.P. 1,589,749, 22.6.26. Appl., 27.2.25).—Alkaline solutions used in gas purification when foul are re-activated by the action of air, the concentration of thiosulphate being maintained above 20 g. per litre. S. I. Levy.

Recovering ammonium sulphate [from coal-gas]. F. F. Marquard (U.S.P. 1,589,809, 22.6.26. Appl., 5.2.25).—The gas is passed through a closed sulphuric acid saturator in which the salt is made as usual and then through a secondary closed washer. The bath in the saturator is periodically neutralised while continuing the passage of the gas so as to remove pyridine. C. Irwin.

Motor fuel. R. A. Carroll and H. L. Elliott (U.S.P. 1,587,899, 8.6.26. Appl., 31.1.20. Renewed 30.10.25).—The fuel consists of aromatic hydrocarbon distillates, free from sulphur, and petroleum distillates, both boiling not above 150°, and also a volatile monohydric alcohol, a readily volatile terpene, naphthalene, and water, the quantity of the last four substances being not more than 7% in all. The product is a homogeneous stable mixture. H. Moore.

Transport and distillation of mineral oils, tar oils, and tars. G. E. Heyl (E.P. 253,436, 15.12.25).—Oil or tar is distilled in the tanks of tank

steamers or barges during transit. The tanks are heat-insulated and subdivided to permit of the concentration of the heating on portions at a time. Heating is effected by a steam coil or surface heat exchanger. Exhaust steam may be utilised in the initial stage, live steam being used after. The vapours may be used to preheat the raw material, being afterwards condensed and stored in separate compartments. H. MOORE.

Motor fuel for internal-combustion engines. F. A. HOWARD, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,589,885, 22.6.26. Appl., 16.8.20).—A motor fuel comprises a knock-producing volatile hydrocarbon liquid containing absorbed ammonia. H. MOORE.

Gasoline-recovery apparatus. H. B. BERNARD, Assr. to SINCLAIR OIL AND GAS Co. (U.S.P. 1,584,504, 11.5.26. Appl., 20.6.24).—In the recovery of gasoline from natural gas by the absorption process, absorbed constituents are separated from the liquid absorbent in an exhausting tower provided with means for promoting contact and heat exchange between gas and liquid. A partition in the lower part of the tower divides it into a heated supply compartment, into which charged absorbent is introduced, and a discharge compartment opening upward into the tower. Liquid may be withdrawn from the supply compartment and discharged into the tower over the contact-promoting devices, and thence conducted to the discharge compartment. Gases and vapours are withdrawn from the upper end of the tower, and liquid from the discharge compartment. H. MOORE.

Apparatus for distilling hydrocarbons. M. J. TRUMBLE (U.S.P. 1,586,130, 25.5.26. Appl., 16.4.23).—Oil heated to a desired volatilisation temperature is delivered to a separator, the discharge of oil from which is regulated in proportion to the feed thereto. The vapour outlet of the separator is provided with a device for regulating the pressure within the separator, and means are provided for converting the expansive heat energy of the vapour products from the separator into mechanical energy. H. MOORE.

Apparatus for refining oil. M. J. TRUMBLE (U.S.P. 1,586,132, 25.5.26. Appl., 2.9.24).—An evaporator for treating petroleum oils comprises a cylinder, shafts supported on bearings projecting from each end of the cylinder, and having a common axis about which the cylinder rotates, and a tight casing enclosing the cylinder. Oil is delivered into the cylinder and oil vapour withdrawn from the casing. The bearings are electrically insulated from the casing, and an electromotive force is established between the ends of the cylinder. H. MOORE.

Continuous refluxing still. M. J. TRUMBLE (U.S.P. 1,586,133, 25.5.26. Appl., 8.9.24).—An apparatus for cracking oils comprises a still, a refluxing coil, to which vapours are passed from the still, and from which condensate is passed to the still, and a condenser, to which vapours are passed from the still. The coil is contained in a chamber to which condensate from the condenser is passed. Surplus liquid from the coil chamber is returned to the still, and the vapours produced in the coil chamber are condensed in a separate condenser. H. MOORE.

Manufacture of light hydrocarbons. L. EDELEANU (U.S.P. 1,586,357, 25.5.26. Appl., 20.1.25. Conv., 17.12.24).—Mineral oils are converted into light hydrocarbons of lower b.p. by depriving them of the constituents soluble in liquefied sulphur dioxide, preparing a double compound of the oil by treating with aluminium chloride, adding gradually the double compound to mineral oil to be converted, and heating the mixture. H. MOORE.

Converting hydrocarbon oils into lower-boiling products. F. A. HOWARD, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,586,994, 1.6.26. Appl., 16.8.22).—Mercury is heated in a closed system to a temperature above 370°, and hydrocarbons are cracked by exposure to the indirect heat of the mercury vapour, the cracked products being withdrawn in vapour form and condensed. H. MOORE.

Apparatus for refining hydrocarbon materials. F. M. FLOYD (U.S.P. 1,587,370, 1.6.26. Appl., 19.5.22).—The apparatus comprises a still and a reflux condenser, the lower end of which communicates with the still. Within the reflux condenser is a coil. An inlet pipe for the material to be distilled enters the condenser at the top, passes down, and is connected to the bottom of the coil, whilst an exit conduit leads from the top of the coil and out through the bottom of the condenser into the still, having an outlet near the bottom of the still. H. MOORE.

Process of treating hydrocarbons. R. CROSS (U.S.P. 1,587,491, 1.6.26. Appl., 9.2.23).—Petroleum oil is vaporised under pressure, and the vapours are passed through a mixture of bentonite and a salt of a metal having an affinity for the sulphur compounds of the oil, the mixture being maintained above the " dry point " of the oil. The vapours are subsequently condensed. H. MOORE.

Treating hydrocarbons and oils. L. B. CHERRY, Assr. to C. & C. DEVELOPING Co. (U.S.P. 1,588,308, 8.6.26. Appl., 11.11.19).—Oils are subjected to a cracking temperature at a cathode while being traversed by an ionising electric current flowing from an anode to the cathode. H. MOORE.

Conversion of high-boiling hydrocarbon oils into lower-boiling oils. P. J. SWEENY, Assr. to STANDARD OIL Co. (U.S.P. 1,589,908, 22.6.26. Appl., 11.1.26).—Oils having a b.p. range similar to that of gasoline are produced by passing the higher-boiling oil continuously in a restricted stream through a zone in which it is brought to conversion temperature. The heated oil is discharged into one of a number of series of connected enlarged chambers, through which it flows at a slower rate, the temperature being maintained, and pressure maintained on the system. Liquid products are

withdrawn from a chamber remote from the one into which the oil first flows from the heating zone. Oil is passed through the chambers until substantial deposits of coke and asphalt are produced in the later chambers of the series, whereupon the oil is passed in the reverse direction through the chambers.
H. Moore.

Process of manufacturing oil of a low boiling point from mineral heavy oils. T. Mii, Assr. to K. Isomura (U.S.P. 1,590,640, 29.6.26. Appl., 19.1.23).—An aluminium hydrosilicate is impregnated with a soluble chloride to produce a catalyst for the manufacture of oils of low boiling point.
H. Moore.

Process of treating hydrocarbons. J. R. McConnell (U.S.P. 1,590,841, 29.6.26. Appl., 27.7.25).—Light hydrocarbons containing a light-sensitive discolouring compound are treated to produce water-white hydrocarbons stable in colour by exposing to light to fix the colour of the discolouring compound and afterwards distilling.
H. Moore.

Refining lubricating-oil stocks. J. W. Weir (U.S.P. 1,581,369, 20.4.26. Appl., 14.5.25).—The oil is treated with sulphuric acid, settled, and most of the sludge removed. The oil stock is treated with a powdered solid adsorbent at a temperature sufficient to decompose the sludge present, liberating sulphur dioxide. Litharge is then added to the mixture, and the oil stock separated. H. Moore.

Process of manufacturing lubricating oils. J. W. Weir (U.S.P. 1,581,370, 20.4.26. Appl., 9.11.25).—The oil stock is treated with sulphuric acid, the sludge removed, and the stock neutralised with alkali, which is then separated from the stock, to which further sulphuric acid is added, and the sludge settled and removed. The stock is then agitated with a powdered solid adsorbent at a temperature below that at which the sludge decomposes. The adsorbent and adsorbed matters are separated from the stock and a further quantity of a powdered solid adsorbent is added, the mixture heated to decompose the remaining sludge, with liberation of sulphur dioxide, and adsorbent and adsorbed material separated from the stock.
H. Moore.

Method of using concentrated decolorising clays for refining lubricating oils. A. Jurrissen, Assr. to Simplex Refining Co. (U.S.P. 1,585,922, 25.5.26. Appl., 31.5.21).—Petroleum lubricating oil is treated first with sulphuric acid, then with decolorising clay, and subsequently neutralised with an alkali, *e.g.*, lime, the oil, acid, and alkali all being free from water. H. Moore.

Preparing a lubricating oil. W. D. Rial (U.S.P. 1,587,859, 8.6.26. Appl., 2.11.25).—A vegetable oil, *e.g.*, castor oil, is treated with a carbocyclic compound capable of increasing its capillarity, *e.g.*, cresol, and then mixed with a mineral oil stock containing a deodorant. A typical lubricant of this nature has the composition: 5% of castor oil, 5% of nitrobenzene, 1% of cresol, and 89% of light lubricating oil. R. B. Clarke.

Desulphurising [mineral] oils. E. M. Johansen, Assr. to Atlantic Refining Co. (U.S.P. 1,587,649, 8.6.26. Appl., 1.5.23).—Oils containing combined sulphur are mixed with oils containing free sulphur and the mixture is treated with alkali plumbite. The sulphur compounds react with the plumbite to form compounds which react with the free sulphur and remove it. E. S. Kreis.

Process for treating petroleum emulsions. M. De Groote, Assr. to W. S. Barnickel & Co. (U.S.P. 1,590,617, 29.6.26. Appl., 23.12.24).—The emulsion is subjected to the action of a water-insoluble salt of a mineral sulphonated oil.
H. Moore.

Continuously separating [acid] petroleum sludge. I. Hechenbleikner and T. C. Oliver (U.S.P. 1,586,801, 1.6.26. Appl., 20.3.23).—Acid sludge is continuously separated into its hydrocarbon and acid constituents by subjecting it to the action of steam under pressure. The components are withdrawn in a mechanically mixed state, and are subsequently physically separated. H. Moore.

Distillation of solid bituminous fuels. H. Rosenthal, Assr. to J. Pintsch A.-G. (U.S.P. 1,592,467, 13.7.26. Appl., 24.3.22. Conv., 19.8.20).—See E.P. 193,276; B., 1923, 342 a.

Gas-purification process. F. W. Sperr, jun., Assr. to Koppers Co. (U.S.P. 1,592,648, 13.7.26. Appl., 8.12.21).—See E.P. 190,116; B., 1924, 164.

Gas-purification apparatus. F. W. Sperr, jun., Assr. to Koppers Co. (U.S.P. 1,592,649, 13.7.26. Appl., 23.5.24. Renewed 3.3.26.)—See E.P. 204,058; B., 1924, 164.

Operating explosion motors. Badische Anilin- & Soda-Fabr., Assees. of W. Gaus and F. Lappe (U.S.P. 1,591,526, 6.7.26. Appl., 29.4.24. Conv., 8.5.23).—See E.P. 215,776; B., 1925, 662.

Separating and purifying [hydrocarbon] oil. W. E. Trent, and Trent Process Corp. (U.S.P. 1,591,728, 6.7.26. Appl., 11.8.20).—See E.P. 167,738; B., 1922, 579 a.

Processes for dressing coal, lignite, and like materials. T. Franz (E.P. 240,799, 15.8.25. Conv., 1.10.24).

Apparatus for supplying coal dust to furnaces by means of compressed air. "Kohlenstaub" G.m.b.H. (E.P. 233,696, 4.5.25. Conv., 7.5.24).

Producing hydrogen (U.S.P. 1,588,860).—See VII.

Nitration of petroleum distillates (U.S.P. 1,588,027).—See XX.

III.—TAR AND TAR PRODUCTS.

Cresylic acid from petroleum distillate. CATLIN.—See II.

PATENTS.

Reclaiming tar and light oil [from aqueous emulsions]. W. F. DIETZSCH (U.S.P. 1,589,374, 22.6.26. Appl., 17.12.24).—The emulsions are heated to a temperature slightly above the boiling point of the light oils, but below that of water. The light oils distil over and are condensed, whilst the tar settles to the bottom of the aqueous layer, and is withdrawn. L. A. COLES.

Transport and distillation of tar oils and tars (E.P. 253,436).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Subsidiary dyes in commercial Agalma Black 10B. W. R. BRODE (Ind. Eng. Chem., 1926, **18**, 708—712).—Spectrophotometric measurements on the exhaust liquors from a 15—16% dyeing of Agalma Black 10B in comparison with dyeings of the pure colour, 1-amino-8-naphthol-3 : 6-disulphonic acid-7-azobenzene, and 2-*p*-nitrobenzeneazo-1-amino-8-naphthol-3 : 6-disulphonic acid show that the red impurities consist almost entirely of 1-amino-8-naphthol-3 : 6-disulphonic acid-7-azobenzene. The characteristic blue colour developed in 10% sodium hydroxide with 2-*p*-nitrobenzeneazo-1-amino-8-naphthol-3 : 6-disulphonic acid was never obtained with these exhaust liquors from the commercial dye. Dyeings stronger than 16% leave an increasing amount of Agalma Black 10B relative to impurity in the mother liquors, although the impurity (1 - amino 8 naphthol - 3 : 6 - disulphonic acid - 7 - azobenzene) has much less affinity for wool than Agalma Black 10B. The amount of dye absorbed by the wool from commercial Agalma Black 10B depends on the concentration in the solution from which the dyeing takes place. In general the amount of impurity present in commercial Agalma Black 10B varies from 1 to 2·5%. The absorption bands of 2-*p*-nitrobenzeneazo-1-amino-8-naphthol-3 : 6-disulphonic acid and 7-benzeneazo-1-amino-8-naphthol-3 : 6-disulphonic acid in neutral or slightly acid solution are almost identical, whilst 2:7-di-(*p*-nitrobenzeneazo)-1-amino-8-naphthol-3:6-disulphonic acid and pure Agalma Black 10B are also very similar in shade and reactions, although the former has a greener shade and more affinity for wool, and consequently would not concentrate in the dye-bath if present as impurity. R. BRIGHTMAN.

See also A., Aug., 789, **Adsorption phenomena** (TESTONI); **Surface actions** (DUBRISAY).

PATENTS.

Manufacture of new [chromium compounds of azo] dyestuffs. SOC. CHEM. IND. IN BASLE (E.P. 235,862, 29.5.25. Conv., 17.6.24).—Dyes which contain chromium and dye animal fibres from an acid bath in fast yellow to orange and brown shades are obtained by treating *o*-hydroxyazo-dyes (obtained from a nitrated *o*-hydroxydiazo-compound and an ester or arylide of acetoacetic acid) with an agent which parts with chromium, such as chromium oxide or its hydrates or salts. Thus 29·5 pts. of the azo-dye from diazotised 4-nitro-2-aminophenol and ethyl acetoacetate are suspended in 900 pts. of water and boiled under a reflux with a quantity of chromium formate corresponding with 22·5 pts. of chromium oxide (Cr_2O_3). When cool the liquid is made alkaline with dilute caustic soda and filtered. The dye separates from the filtrate as a yellow-brown powder dyeing wool from a sulphuric acid bath in fast reddish-yellow shades. The dye from 4-nitro-2-aminophenol-6-sulphonic acid and acetoacetanilide similarly yields an olive-brown powder dyeing wool in reddish-yellow shades, whilst that from 6-nitro-2-aminophenol-4-sulphonic acid and acetoacetanilide yields a brown powder dyeing reddish-yellow shades on wool. Azo-dyes from 4-nitro-2-aminophenol-6-carboxylic acid similarly yield dyes giving greenish-yellow shades on wool, whilst brown shades are obtained when the initial azo-dye is prepared from a nitrated *o*-hydroxydiazo-compound of the naphthalene series. Other arylides of acetoacetic acid may be used in place of acetoacetanilide. R. BRIGHTMAN.

Manufacture of dyestuffs capable of being chromed. SOC. OF CHEM. IND. IN BASLE (E.P. 251,266, 15.4.26. Conv., 25.4.25).—The dyes described in E.P. 214,629 (cf. U.S.P. 1,521,206; B., 1925, 165) can be obtained in much higher yield by conducting the reduction with sulphide in the presence of an organic compound containing hydroxyl groups, such as dextrose, sucrose, glycerol, glycol, tannin, gallic acid, or sulphite-cellulose waste lye. Thus the concentrated suspension of the dye obtained from 150 pts. of α-naphthol and 295 pts. of 1-amino-2-naphthol-4-sulphonic acid is mixed, while stirring, with 25 pts. of dextrose or glycerol, 145 pts. of 30% sodium hydroxide, and 360 pts. of sodium sulphide crystals. The mixture is stirred at the ordinary temperature until the sulphide has disappeared, when it is diluted with water and the dyestuff salted out. R. BRIGHTMAN.

Manufacture of colouring matters and dyeings therewith. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 253,659, 16.4.25).—Chromium derivatives soluble in water which have the property of giving direct dyeings of excellent fastness on cotton or other cellulose fibres, natural or artificial, are obtained from direct azo dyes containing groups which enable them to be chromed, for instance, azo dyes containing two radicals of an *o*-hydroxy-carboxylic acid in their molecule. The dyes may be mixed with a soluble chromium salt and the mixture used for dyeing without further treatment. Alternatively the dye-bath may be prepared from the dyes and the chromium salts separately. Chromium fluoride, acetate, oxalate, or formate may be used, or complex chromium salts containing chromium both as an anion and a cation. For instance 10 pts. of the azo dye from 4 : 4′-diamino-

stilbene-2 : 2′-disulphonic acid and 2 mols. of salicylic acid, dissolved in 400 pts. of water with 5 pts. of sodium acetate crystals, are boiled for about 3 hrs. with 100 pts. of a chromium salt solution, containing about 10 pts. of chromic oxide (Cr_2O_3). The solution is filtered if necessary and the dye salted out. For dyeing, 100 pts. of cotton are treated in a hot bath containing 3 pts. of the chromium compound with a little Glauber's salt or common salt for an hour, or manipulated for several hours at the ordinary temperature. Fast orange shades are obtained. Examples of other methods of preparing and applying the dye are given. The dye from 2 mols of *p*-aminosulphosalicylic acid (obtained by sulphonating *p*-aminosalicylic acid with 23% oleum) and 1 mol. of 5 : 5′-dihydroxy-2 : 2′-dinaphthylcarbamide-7 : 7′-disulphonic acid gives red shades, the analogous dyes from 5 : 5′-dihydroxy-2 : 2′-dinaphthylamine-7 : 7′-disulphonic acid and from *o*-amino-*p*-sulphosalicylic acid give violet shades. Violet shades are also obtained with the dye from 1 mol. of benzidine-2 : 2′-disulphonic acid and 2 mols. of 2-(4′-hydroxy-3′-carboxyphenyl)-amino-5-naphthol-7-sulphonic acid and with the dye obtained from 1 mol. of the latter acid and 1 mol. of 4-amino-4′-hydroxy-5′-carboxydiphenylamine-2-sulphonic acid. Blue shades are obtained with the dye from 1 mol. of the aminonaphtholsulphonic acid derivative and 1 mol. of 4 : 4′-diaminostilbene-2 : 2′-disulphonic acid. Yellow to orange shades are obtained with the dyes from 1 mol. of benzidine and 2 mols. of salicylic acid or *m*-cresotic acid and from 2 mols. of salicylic acid and 1 mol. of *oo*′-dianisidine or *pp*-diaminodiphenylcarbamide. R. Brightman.

Manufacture of monoazo-dyestuffs or intermediate compounds for manufacture of disazo or polyazo-dyestuffs. A. G. Bloxam. From Chem. Fabr. Griesheim-Elektron (E.P. 253,763, 23.9.25). —Monoamines of the diphenyl series, especially their nitro- and acetamido-substituted derivatives, are diazotised and coupled with a 1-aryl-5-pyrazolonesulphonic acid, a naphtholsulphonic acid, or an aminonaphtholsulphonic acid, giving monoazo-dyes which possess valuable properties, especially fastness to milling, and are also useful intermediates for disazo- or polyazo-dyes. Thus *pp*′-dichloroaminobenzidine, m.p. 95—96°, diazotised at 5—10° and coupled alkaline with *p*-sulpho-*o*-chlorophenylmethylpyrazolone gives a yellow wool dye, with 2 : 6-naphtholsulphonic acid a reddish-brown dye, and with 2-phenylamino-8-naphthol-6-sulphonic acid a brown dye. Other substituted amino-compounds of monoamines of the diphenyl series may be used, with the exception of monoamines containing the substituent group NHR, where R is an aryl group. Sulphonated monoamines of the diphenyl series may also be used, but in this case the coupling component should be unsulphonated. R. Brightman.

Manufacture of indigoid dyestuffs containing sulphur. Farbw. vorm. Meister, Lucius, & Brüning (E.P. 251,293, 7.4.25. Conv., 9.4.24).—Indigoid dyes containing sulphur are obtained by heating an *O*-acyloxythionaphthen with a 2:3-diketodihydrothionaphthen-α-anil, either in the presence or absence of a solvent or diluent. The new dyes are distinguished by the variety and fastness of their shades. The *dye* from 3-acetoxythionaphthen (3·8 pts.) and 5-chloro-7-methyl-2:3-diketodihydrothionaphthen-α-(*p*-dimethylamino)anil (6·6 pts.) at 150° gives bluish-red shades on cotton whilst the *dye* obtained by boiling 4·2 pts. of the same anil with 3 pts. of 5:6-benzo-3-acetoxythionaphthen and 300 pts. of acetic acid until there is no further separation of dye, dyes cotton in the vat a violet shade. R. Brightman.

Dyestuffs and dyeing of acetyl silk. Scottish Dyes, Ltd., E. G. Beckett, J. Thomas, and R. Tonkin (E.P. 253,584, 16.3.25. Addn. to 231,260; cf. B., 1925, 393).—In place of the alkali sulphide used in the original patent, an alkaline-earth sulphide may be used, especially calcium hydrogen sulphide. Thus in place of 168 pts. of sodium sulphide crystals, 73 pts. of calcium hydrogen sulphide may be used. R. Brightman.

Dye compositions [for dyeing silk]. J. M. Matthews, Assr. to Glorient, Inc. (U.S.P. 1,588,482—4, 15.6.26. Appl., [A] 29.5.24, [B] 8.11.24, [C] 2.9.25).—Non-hygroscopic powders which readily yield solutions suitable for dyeing silk contain (A) an acid dye not precipitated in the presence of aluminium sulphate and potassium bitartrate, a substance which supplies free acid when dissolved in water, and a levelling agent; (B) a soluble organic acid, a levelling agent, and an acid dye not precipitated by the acid and the levelling agent; or (C) an acid dye, sodium bifluoride, and a levelling agent. L. A. Coles.

Manufacture of vat colouring matters. J. Y. Johnson. From Badische Anilin & Soda Fabr. (E.P. 254,476, 2.6.25).—See F.P. 599,038; B., 1926, 577.

Production of new insoluble azo dyestuffs in substance or on the fibre. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 254,578, 29.10.25).—See U.S.P. 1,576,322; B., 1926, 432.

Manufacture of diaminodiaryldialkylmethanes. B. Homolka, Assr. to Grasselli Dyestuff Corp. (U.S.P. 1,591,384, 6.7.26. Appl., 19.9.23. Conv., 29.9.22).—See E.P. 204,722; B., 1924, 412.

Preparing halogenated oxythionaphthens. E. Hoffa, Assr. to Grasselli Dyestuff Corp. (U.S.P. 1,592,210, 13.7.26. Appl., 5.4.24. Conv., 28.4.23).—See E.P. 215,021; B., 1925, 438.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

"Immune" [cotton] yarn. M. Fahrländer (Textilber., 1926, 7, 463—464).—Immune cotton yarn (cf. E.P. 246,609; B., 1926, 270) may be subjected to processes of singeing, de-sizing, scouring, bleaching, and mercerising as employed for cotton. It has dyeing properties similar to those of cellulose

acetate silk and a special range of Imacol dyes capable of dyeing immune yarn in shades fast to light and washing but having no affinity for cotton is now available. Imacol dyes (Sandoz) may be used with direct cotton dyes in the same dye-bath.

A. J. HALL.

Influence of curtailed time of heating up on the constitution of sulphite pulp. O. ROUTALA and J. SEVON (Zellstoff u. Papier, 1926, 6, 257—259).—Much can be done by suitable manipulation of the temperature at different stages to shorten the total time of digestion of sulphite pulp, particularly the very prolonged operation of the Mitscherlich process. Experimental digestions showed that the early stage of heating-up to the boil, which is generally extended over 4 hrs., can be curtailed to 20 min. if, after reaching 105°, the charge be held at this for 1 hr., then again at 110° for 2 hrs., and finally at 120° for 1 hr. before proceeding to the digestion proper at 130—135°. The object of the slow heating-up is to give time for the penetration of the wood by the liquor, but in the region slightly above 100° the velocity of the chemical reactions is still low, whilst the diffusion velocity is very much greater than at the usual heating-up stage. Thus provided the chemical reactions are not unduly hastened by proceeding too rapidly to the full digestion temperature, no trouble is to be feared from defective penetration by omitting the slow heating-up stages.

J. F. BRIGGS.

Loading and sizing of paper. H. ROSCHIER (Papierfabr., 1926, 24, 348—350, 363—365, 384—388).—Experiments on clay-retention efficiencies were carried out by means of sheets made on the Lampen mould from unbeaten sulphite pulp with which the clay was mixed with various additions. In the presence of 2% of aluminium sulphate only, the yield of clay in the paper decreases as the dilution of the pulp is increased. Other conditions being constant, the yield of clay increases with the proportion of aluminium sulphate until the latter reaches 4—5% of the dry weight of cellulose, depending probably on the precipitation of the hydroxide. Excess of aluminium sulphate, that is of acidity, is unfavourable, but if acidity be reduced by the addition of sodium hydroxide the yield of clay in the paper with a constant proportion of aluminium sulphate increases to a maximum when the p_H value is 5·6. If the p_H value be maintained constant at 5·5 by the addition of sodium hydroxide while the proportion of aluminium sulphate is increased, the maximum efficiency of clay retention is reached between 6 and 8% of aluminium sulphate on the cellulose. Under these conditions increased retention efficiencies are obtained with increased proportions of clay added, up to about 30% of clay, above which the percentage retention quickly falls. Starch when added as a jelly largely increases the retention of clay, but a clear filtered solution of starch has no effect. Resin size has a favourable influence on the retention of clay, so long as the proportion of aluminium sulphate is quite low, but with larger proportions of alum the effect of size is not nearly so pronounced as that of plain sodium hydroxide. The retention of clay by the pulp is primarily and mainly a mechanical filtration process but colloidal influences play a minor part. The favourable influence of beating is most marked. High dispersion of the clay particles is unfavourable to retention, and in fractionated clays the coarser fractions give the highest yields in the paper. This influence of the size of particle is, however, largely mitigated by the beating of the pulp, so that finely beaten pulps favour the retention of the finer clay particles. The retention efficiency of talc is very much better than that of clay, especially with little or no alum present. As the percentage of aluminium sulphate is increased the clay responds much more favourably than the talc and the difference becomes less marked.

J. F. BRIGGS.

Errata.—B., 1926, April 16, p. 267, col. 2, line 24 from bottom, *for* "1926, 1, 1—6" *read* "1926, 7, 1—6"; also p. 268, col. 1, lines 3 and 6 from top, *for* "*iso*saccharic" *read* "*iso*saccharinic."

See also A., Aug., 781, **x-Ray investigations on cellulose** (HERZOG). 793, **Comparison of heat effect of swelling and mercerisation of cellulose with absorption and x-Ray spectrographic experiments** (KATZ); **Swelling of cellulose** (HERZOG).

PATENTS.

Decolorising solutions of cellulose derivatives. AKT.-GES. F. ANILIN-FABR. (E.P. 245,078, 12.11.25. Conv., 23.12.24).—A 10% solution of coloured (dyed) celluloid film in ether–alcohol, acetone, ethyl acetate, etc., is stirred with a substance containing silicic acid and having adsorbent surface activity, *e.g.*, fuller's earth or Florida bleaching earth. The quantity of decolorising agent may be 20% of the weight of the film, and after stirring for 2—3 hrs. the earth is allowed to settle for a similar length of time and the decolorised solution clarified by filtration. Alternatively a 5% solution of the coloured celluloid may be forced through a layer of fuller's earth, the solution coming through decolorised.

J. F. BRIGGS.

Process of making acetylated nitrocellulose. H. P. BASSETT and T. F. BANIGAN, Assrs. to MEIGS, BASSETT & SLAUGHTER, INC. (U.S.P. 1,586,437, 25.5.26. Appl., 11.5.23).—Nitrocellulose of high nitrogen content is dissolved in a solvent adapted under the influence of a suitable catalyst to acetylate the nitrocellulose; the solution cooled below 15°, a suitable catalyst is added to the cooled solution, and the temperature is maintained below 15° until acetylnitrocellulose is formed. J. F. BRIGGS.

Nitrocellulose composition. E. R. LITTMANN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,589,608, 22.6.26. Appl., 17.10.25).—The composition consists of nitrocellulose and tri-n-butyl phosphate. C. IRWIN.

Viscose artificial silk fibres. ERSTE BÖHMISCHE KUNSTSEIDEFABRIK A.-G. (F.P. 600,309, 3.7.25).—

Capillary-active materials such as gum, mucin, proteins, pectin, betaine, urethanes, alcohols, fatty acids, etc., are added to the precipitation baths.

B. W. CLARKE.

Low-temperature explosion process of disintegrating wood and the like. W. H. MASON (U.S.P. 1,586,159, 25.5.26. Appl., 6.7.25).—Lignocellulose material is introduced into a high-pressure chamber together with a moist fluid, and a compressed gas is introduced into the chamber at a pressure higher than that of the moist fluid. The contents of the chamber are discharged through a relatively restricted outlet, whereby the material is disintegrated without being subjected to temperatures which would char or discolour it. J. F. BRIGGS.

Improving artificial silk, staple fibres, and textiles made therewith. H. KARPLUS (U.S.P. 1,591,922, 6.7.26. Appl., 2.6.24. Conv., 11.1.24).—See E.P. 234,618; B., 1925, 707.

Utilising the skins of chondropterygians and plagiostomes. A. EHRENREICH and K. BENDIXEN (U.S.P. 1,592,189, 13.7.26. Appl., 4.6.24. Conv., 7.6.23).—See E.P. 235,050; B., 1925, 626.

Nitration of petroleum distillates (U.S.P. 1,588,027).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Comparison of "wetting-out" agents [for textile processes]. J. AUERBACH (Textilber., 1926, 7, 681—685).—The wetting-out powers of various commercial products were compared by determining the times required for a square of dry raw woollen fabric to sink when placed on the surface of their aqueous solutions. Oranit, Nekal A pdr., and Neomerpin N are particularly efficient wetting-out agents, sodium carbonate, alcohol, Turkey-red oil, Eufullon, and Verapol being considerably less effective. Most of the wetting-out agents were more effective in hot solutions. A. J. HALL.

Indigosol O [in dyeing and printing]. G. FRIEDLÄNDER (Textilber., 1926, 7, 697—698).—Details of numerous large-scale processes for printing and dyeing indigo effects on fabrics by means of Indigosol O (cf. Indigosol DH, Vaucher and Bader, B., 1924, 864) are described. Fabric is satisfactorily dyed by slop padding with a solution containing 80 g. of Indigosol O, 835 g. of water, 50 g. of 8% gum tragacanth, 20 g. of a 10% β-naphthol solution, and 15 g. of sodium nitrite, drying in a hot-flue or on drying cylinders, and passing the dried fabric during 10—30 sec. through a developing bath at 20° containing 20 c.c. of sulphuric acid of 66° B. (*d* 1·84) per litre and then thoroughly washing it free from acid. It is recommended to use 1·2, 1·5, 2·0, 2·5, and 3·0% of sodium nitrite with 1—4, 4—8, 9—11, 12—15, and 16—20% of Indigosol O respectively, the ratio between the amounts of these substances used in the padding liquor being of considerable importance. The presence of β-naphthol increases the stability of the padding liquor. An addition of an oxidisable acid such as formic acid to the developing bath reduces the liberation of obnoxious nitrogen oxide vapours, and drying of the padded fabric may be less complete if salt or Glauber's salt is added to the padding liquor. Very thick cotton and half-linen fabrics are not padded but treated in a jig for ½ hr. at 20—25° with a solution containing 40 pts. of Indigosol O, 20 pts. of sodium nitrite, 180 pts. of calcined sodium sulphate, 5 pts. of Turkey-red oil, and 1000 pts. of water, then evenly squeezed, entered directly into the acid developing bath, afterwards neutralised with a dilute solution of sodium carbonate, washed, and dried. A satisfactory "steam" process consists of padding fabric with a solution containing 100 pts. of Indigosol O, 705 pts. of water, 50 pts. of an 8% solution of gum tragacanth, 15 pts. of ammonium thiocyanate, 80 pts. of a 10% solution of sodium chlorate, and 50 pts. of a 1% solution of ammonium vanadate, or 80 pts. of Indigosol O, 20 pts. of ammonium chloride (or ammonium thiocyanate), 10 pts. of sodium chlorate, 10 pts. of sodium ferrocyanide, and 880 pts. of a suitable thickening, then drying in a hot flue (or on drying cylinders having the first few cylinders lapped with fabric), and steaming in a Mather-Platt or in a closed steaming chamber (particularly suitable for the last-named padding solution) for 2—15 min. Indigo effects may also be obtained by the usual methods for producing aged Aniline Black using a padding liquor containing 100 g. of Indigosol O, 555 g. of water, 50 g. of glycerol, 10 g. of Turkey-red oil, 25 g. of a 50% solution of gum, 200 g. of ammonium chlorate of 15° B. (*d* 1·115), 40 g. of ammonium oxalate, and 40 g. of a 1% solution of ammonium vanadate. It is possible to use padding liquors containing Indigosol O and Katanol W or tannic acid or direct cotton dyes or chrome colours, and fabrics printed with Indigosol O pastes may be overprinted with Aniline Black.

A. J. HALL.

Griesheim Naphthol AS red pigments in dyeing and printing. W. KIELBASINSKI (Textilber., 1926, 7, 611—612).—Rapid Fast Red B is a mixture of 5-nitro-2-aminoanisole (the nitrosoamine), Fast Red Base B, and Naphthol AS; Rapid Fast Red BB is a mixture of 5-nitro-2-aminoanisole (nitrosoamine), Fast Red Base B, and Naphthol AS.BS; Rapid Fast Red 3 GL is a mixture of *o*-nitro-*p*-chloroaniline (nitrosoamine) and Naphthol AS; Rapid Fast Red GL is 3-nitro-*p*-toluidine (nitrosoamine) and Naphthol AS; Rapid Fast Red GZ is 2:5-dichloroaniline (nitrosoamine) and Naphthol AS; Rapid Fast Orange RG is *o*-nitroaniline (nitrosoamine) and Naphthol AS; Rapid Fast Blue is dianisidine (nitrosoamine) and Naphthol AS (cf. Rowe and Corbishley; B., 1925, 797). In dyeing loose cotton with Naphthol AS colours, maximum fastness to rubbing is obtained only when the fibres prepared with naphthol are thoroughly washed with water (an alkaline solution of sodium chloride has been recently recommended) before coupling with the diazo-component, and the resulting dyeing should

be after-treated with a hot solution of caustic soda. Naphthol AS couples less rapidly than β-naphthol with diazo-compounds. A. J. HALL.

Printing nitrosophenol dyestuffs [on fabrics]. P. WENGRAF (Textilber., 1926, 7, 447—448).—Deep dark green, reddish mode, and dark tobacco-brown shades are obtained by printing fabric prepared with β-naphthol with pastes containing an organic acid, sodium nitrite, and a salt of iron, nickel, or chromium respectively, the fabric being afterwards steamed for 5 min. in an air-free Mather-Platt. Suitable printing pastes contain 820 g. of an acetic acid and gum tragacanth thickening, 100 g. of a solution of iron acetate of 14° B. (*d* 1·108), nickel acetate of 16° B. (*d* 1·125), or chromium acetate of 11° B. (*d* 1·083), 30 g. of glycerol, and 50 g. of sodium nitrite. Attempts to prepare more stable printing pastes by using ammonium salts instead of acetic acid were unsuccessful, the rate of dissociation of such salts during steaming being too slow. A 2·2% solution of β-naphthol is suitable for preparing the fabric before printing. The resulting dyeings (colour lakes) have a good affinity for basic dyes. Coloured discharges may be obtained by means of sodium hyposulphite and certain basic, vat, and sulphur dyes. A. J. HALL.

Comparison of maize and potato starches for textile finishing. W. EKHARD (Z. Spiritusind., 1926, 49, 196—197).—Potato starch contains much less fat and protein than maize starch and yields more viscous aqueous solutions. The viscosities of 5% solutions of potato, maize, and wheat starches are as 3·57 : 1·17 : 1·00, and a solution containing 150 g. of maize starch and 2850 g. of water has nearly the same viscosity as a similar solution containing only 100 g. of potato starch to 2900 g. of water. Aktivin (cf. Feibelmann, B., 1926, 484) attacks potato starch more rapidly than maize starch, and textile finishing pastes prepared by the action of Aktivin on potato starch are about one-half as viscous as similar pastes prepared from maize starch. Biolase and Diastafor, Degomma, and Novo Fermasol liquefy potato starch much less rapidly, equally, and slightly less rapidly respectively than maize starch. Potato starch has better binding properties when used in the weighting of textile materials. It is very difficult to prepare solutions of maize starch free from gelatinised "lumps" which are undesirable in finishing pastes. A. J. HALL.

PATENTS.

Mordanting and dyeing of wool. C. S. BEDFORD (E.P. 253,662, 23.4.25).—Wool is mordanted by means of a dichromate and sodium thiosulphate, so that the dichromate is completely reduced to chromium hydroxide, since a mordant in this state gives the fastest shades to light, milling, and potting with suitable mordant dyes; in most cases the resulting shades are brighter and deeper than similar shades on wool mordanted by the usual methods. For example, 500 lb. of wool are treated for 20 min. at 90—93° with a solution containing 10 lb. of sodium dichromate, then 15 lb. of sulphuric acid are added, and, after a further 20 min., 5 lb. of sodium thiosulphate and $\frac{1}{4}$—$\frac{1}{2}$% (calc. on the weight of wool) of borax or $\frac{1}{2}$—1% of sodium acetate; the mordanting is completed within a further 30 min. The addition of borax or sodium acetate prevents the deposition of sulphur. Subsequently the mordanted wool is treated in the same or a separate bath at 60—93° with a mild alkali such as sodium carbonate or ammonia whereby the mordant in the wool is completely converted into chromium hydroxide. The wool may frequently be dyed afterwards in the exhausted mordanting liquor. A. J. HALL.

Printing of fabrics. C. DREYFUS, Assr. to AMER. CELLULOSE & CHEMICAL MANUF. CO., LTD. (U.S.P. 1,588,951, 15.6.26. Appl., 20.9.24).—Union fabrics containing fibres of a cellulose derivative are printed with a mixture containing a solvent for the cellulose derivative, an inert powder, and a thickening agent, the printed fabric being maintained at a temperature not exceeding 125° until at least a portion of the cellulose derivative in the printed parts is removable by washing with water. A. J. HALL.

[Apparatus for] treatment of textile goods with liquids. A. W. SCHMID-KOECHLIN (E.P. 245,783, 7.1.26. Conv., 9.1.25).

[Machines for] calico printing. A. CROMPTON (E.P. 254,441, 27.4.25).

Dyeing of acetyl silk (E.P. 253,584).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Analysis of sulphur dioxide in the presence of excess air. E. D. RIES and L. E. CLARK (Ind. Eng. Chem., 1926, 18, 747).—To determine a small quantity of sulphur dioxide in air, a measured volume of the air is passed through a 4-bulb Mitscherlich absorber containing 10 c.c. of 10% sodium hydroxide solution 0·002*M* in stannous chloride. The solution is washed into a flask, diluted to 50 c.c., treated with 50 c.c. of 12*N*-hydrochloric acid and 2 c.c. of carbon tetrachloride as indicator, cooled, and titrated with 0·003*M*-potassium iodate solution until the pink colour in the indicator disappears. From the amount of iodate used is deducted that consumed in a blank test on the reagents and the remainder is calculated to sulphur dioxide. Low and erratic results were obtained with the following absorbents: solutions of iodine, sodium peroxide, sodium hypochlorite, acid potassium dichromate, acid permanganate, iodine and sodium hydrogen carbonate, and solid lead peroxide. The use of glycerol and sodium hydroxide gave poor results when only traces of sulphur dioxide were present; 5% of glycerol did not prevent oxidation and 50% gave too high a blank (cf. Haller, J.S.C.I., 1919, 52 T, and Berl, B., 1921, 580A). A. R. POWELL.

Absorption and desorption of ammonia in a coke-packed column. T. K. SHERWOOD and

A. J. Kilgore (Ind. Eng. Chem., 1926, 18, 744—746).—The capacity coefficient of a coke-packed column for the absorption of ammonia from an ammonia–air mixture by water and the stripping of ammonia from aqueous solution by air was determined. The rate of liquor feed was maintained constant and the gas and liquor temperatures approximately so, but the gas velocity was varied. The capacity coefficient, *i.e.*, g. of ammonia diffusing per hr. per c.c. of packed column per 1 atm. difference in partial pressures of ammonia in gas and over solution, increased with the gas velocity in a linear relationship. Values for absorption and desorption were identical within the limits of experimental error and it is thought therefore that data obtained for the two processes should be interchangeable. C. Irwin.

Separation of copper and ferrous sulphates by crystallisation. G. Agde and H. Barkholt (Z. angew. Chem., 1926, 39, 851—855).—From a phase-rule study of the system copper sulphate–ferrous sulphate–water it is shown that the triclinic mixed crystal phase consisting of 95—100% $CuSO_4,5H_2O$ and 5—0% $FeSO_4,5H_2O$ does not exist, the solid phase in equilibrium with a solution containing 35—40% of copper sulphate and less than 6% of ferrous sulphate being pure $CuSO_4,5H_2O$. From solutions richer in iron the monoclinic mixed crystal phase containing 61—62% $CuSO_4,7H_2O$ crystallises out on evaporation or cooling. On the ferrous sulphate side a continuous series of monoclinic mixed crystals containing $CuSO_4,7H_2O$ and $FeSO_4,7H_2O$ in molecular ratios varying from 1 : 50 to 1 : 0·66 crystallises out. The latter is identical with the double salt described by Pisani as $3CuSO_4,7H_2O,2FeSO_4,7H_2O$; it appears, however, not to be a true double salt, but simply the limiting solid solution. Recrystallisation of mixed crystals of any composition invariably yields crystals poorer in copper sulphate and a correspondingly richer mother liquor. For technical purposes pure copper sulphate can be obtained by evaporation and cooling only from solutions containing 35—40% of copper sulphate and less than 6% of ferrous sulphate and even then only when the solution is thoroughly agitated during cooling. By isothermal evaporation of solutions of copper sulphate containing only small proportions of iron, more than half the copper salt may be obtained in the form of fine crystals between 50° and 56°. When, however, the composition of the solution reaches the region of the mixed crystal phase, then the copper sulphate is contaminated with these crystals. The crystallisation operation may be controlled by determinations of the ferrous sulphate content of the solutions by titration with permanganate combined with a determination of the specific gravity. Graphs are given showing the solubility isotherms and cooling curves of the system and the relation between sp. gr. and composition of the solution. A. R. Powell.

Calcination rates of limestone. W. A. Gilkey (Ind. Eng. Chem., 1926, 18, 727—728).—Curves showing the relation between the temperature of calcination of limestone and the time required for complete conversion into oxide, and between the temperature of calcination and the percentage conversion in 1 min. are constructed, based on the results obtained by heating small quantities of limestone in a current of air in an electric furnace. In all cases the rate of conversion into oxide varied with the pressure of carbon dioxide, but, under comparative conditions, it was found that the rate of complete conversion at 750° is about 2·3 times, at 850° about 12 times, and at 1000° about 60 times that at 700°. A. R. Powell.

See also A., Aug., 787, **Is orthoboric acid volatile in steam ?** (Nasini). 791, **Rapid method of dialysis, and application to preparation of dialysed ferric hydroxide** (Fabre and Penau). 798, **Thermal dissociation of calcium and magnesium carbonates** (Bäckström). 804, **Electrolytic formation of ammonium persulphate** (Essin).

Hydrogen from water-gas. Evans and Newton.—See II.

Electrolysis of sodium chromate with a mercury cathode. Stscherbakov and Essin.—See XI.

Patents.

Production of concentrated nitric acid from waste acids. H. N. Lentz, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,590,043, 22.6.26. Appl., 20.12.21).—Waste acid from nitration, free from aromatic compounds and containing more nitric acid than nitrososulphuric acid, is denitrated by steam in a packed tower. Concentrated nitric acid is condensed hot from the exit gases. The remainder of these, consisting largely of nitrogen peroxide, passes on through a cooling coil to further towers in which it is converted into dilute nitric acid which is returned to the process. C. Irwin.

Apparatus for producing sulphurous acid gases. I. Hechenbleikner, Assr. to Chem. Construction Co. (U.S.P. 1,590,622, 29.6.26. Appl., 1.4.24).—The apparatus consists of a combustion chamber for holding liquid sulphur, with means for introducing an oxidising fluid, including a central inlet and a deflecting hood to direct the fluid downward on to the surface of the sulphur, the level of which is varied according to the concentration of gaseous sulphur dioxide it is desired to produce. H. Royal-Dawson.

Ammonia distillation. J. S. Unger (U.S.P. 1,581,367, 20.4.26. Appl., 22.12.24).—A distillation apparatus comprises a distilling column, a lime vessel, and a grate for supporting lime in the bottom of this vessel. Liquor may be passed from the distilling column to the bottom of the lime vessel, and up through the grate and the lime. The liquor from the lime vessel above the lime charge may be delivered to a lime leg, and by an overflow from the lime leg back to the column. H. Moore.

Purification of gases for ammonia synthesis. G. Claude, Assr. to Lazote, Inc. (U.S.P. 1,589,628,

22.6.26. Appl., 25.3.22. Conv., 25.3.21).—Carbon monoxide and oxygen are removed from the nitrogen–hydrogen mixture to a practically complete extent by passing the gases over a heated catalyst. The process is carried on under pressure in an apparatus resembling the converter in which ammonia synthesis is performed. C. IRWIN.

Process of producing alkali aluminates. RHENANIA VEREIN CHEM. FABR. A.-G. (E.P. 241,232, 12.10.25. Conv., 13.10.24).—Pulverised bauxite (100 pts.) containing 55% Al_2O_3 and 3% SiO_2 is mixed with 82 pts. of sodium sulphate and 5·5 pts. of calcium carbonate and heated to incandescence at 1800° in a rotary furnace heated with gas, steam being passed in at the same time. H. ROYAL-DAWSON.

Method of treating limestone. F. C. MATHERS and H. B. BRISCOE (U.S.P. 1,588,253, 8.6.26. Appl., 14.7.25).—Dolomitic limestone is mixed with 0·6% by weight of potassium chloride or sodium sulphate and calcined for 2 hours at 1100°. The quicklime obtained is ground and converted into the dry hydrate by addition of a calculated quantity of water. R. B. CLARKE.

Production of calcium arsenate. H. M. SCHLEICHER, Assr. to AMER. METAL CO., LTD. (U.S.P. 1,588,499, 15.6.26. Appl., 11.11.24).—Dry unslaked lime is added to a heated solution of alkali arsenate. H. ROYAL-DAWSON.

Method of making arsenates. H. P. BASSETT and E. C. LATHROP (U.S.P. 1,588,691, 15.6.26. Appl., 10.4.24).—Arsenious compounds are oxidised to arsenates by treatment with chlorine in the presence of a base forming a soluble chloride and an almost insoluble arsenate. The latter is subsequently treated with a compound, the cation of which forms a more soluble arsenate or a mixture of more soluble arsenates, and the anion of which combines with the base of the insoluble arsenate to form an insoluble compound. H. ROYAL-DAWSON.

Method of making potassium ferricyanide. G. BARSKY, Assr. to AMER. CYANAMID CO. (U.S.P. 1,589,041, 15.6.26. Appl., 2.10.25).—A small amount of alkali is added to a solution of potassium ferrocyanide, which is afterwards subjected to oxidation. H. ROYAL-DAWSON.

Separation of sodium chloride from mixed salts. C. E. DOLBEAR (U.S.P. 1,589,519, 22.6.26. Appl., 25.4.25).—The mixed salts are subjected to the action of cold water for a limited period, after which the undissolved salts are separated from the brine. H. ROYAL-DAWSON.

Reclaiming spent pickling solutions. H. S. MARSH and R. S. COCHRAN, Assrs. to AMER. COPPERAS CO. (U.S.P. 1,589,610, 22.6.26. Appl., 8.7.25).—Fresh acid is added to the solution of iron salts in weak sulphuric acid and the ferrous sulphate removed from solution. C. IRWIN.

Process for making anhydrous zinc chloride. N. C. CHRISTENSEN (U.S.P. 1,590,229, 29.6.26. Appl., 7.6.23).—Dry lead chloride is heated with metallic zinc. H. ROYAL-DAWSON.

Increasing the density of finely-divided material [soda ash]. E. T. ASPLUNDH, Assr. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,590,795, 29.6.25. Appl., 22.9.24).—Light pulverised hydrated soda ash is subjected to heavy pressure to form it into solid briquettes having the desired density, and the briquettes are pulverised to a granular powder of the desired degree of fineness. H. ROYAL-DAWSON.

Preparing cyanogen chloride. R. V. HEUSER, Assr. to AMER. CYANAMID CO. (U.S.P. 1,588,731, 15.6.26. Appl., 25.11.24).—A solution of cyanide is passed downwards in counter-current to chlorine. H. ROYAL-DAWSON.

Gas [hydrogen] manufacturing machine. C. F. ADAMS (U.S.P. 1,588,153, 8.6.26. Appl., 11.1.22).—The apparatus is especially suited to the manufacture of hydrogen. The solid reagent after being ground in a receiving chamber falls through a valve-controlled hopper into a cylindrical mixing chamber provided with a mechanical stirrer and a waste outlet at the bottom. The gas passes upwards into an outlet tube and flows through a condenser, cooled by air and water, into a gas holder. R. B. CLARKE.

Method of producing hydrogen. C. C. VAN NUYS, Assr. to AIR REDUCTION CO. (U.S.P. 1,588,860, 15.6.26. Appl., 2.8.21).—A mass of ignited carbon is subjected to the action of steam and oxygen in a gas producer, and the resulting gas is separated into its constituents by liquefaction, the oxygen for the producer and the refrigerating agent for the liquefaction process being thereby produced at the same time. H. ROYAL-DAWSON.

Production of oxygen-free nitrogen. F. J. METZGER, Assr. to AIR REDUCTION CO. (U.S.P. 1,588,258, 8.6.26. Appl., 15.1.26).—To a mixture of nitrogen and oxygen is added a proportion of combustible gas corresponding to the oxygen, and the mixture is led over a catalyst, preferably carborundum, in a special nickel-steel vessel. The catalyst is heated electrically to 400° and the gases are introduced at a pressure of 1000—2000 lb. per sq. in. The combustion products are liquefied at this pressure directly the gases pass away from the reaction zone, leaving a residue of pure nitrogen. E. S. KREIS.

Production of pure table or the like salt from impure salt. N. V. MATECHU MAATS. TOT EXPLOIT. VAN CHEM. UITVINDINGEN, Assees. of M. KRUGER and S. R. UNKEL (E.P. 237,867, 18.4.25. Conv., 2.8.24).—See F.P. 593,279; B., 1926, 156.

Recovering lithium salts from phosphate minerals containing lithium. H. WEIDMANN, Assr. to ALLIED PROCESS CORP. (U.S.P. 1,591,196,

6.7.26. Appl., 24.11.24. Conv., 12.12.23).—See G.P. 413,722; B., 1925, 757.

Process of [mechanically] treating asbestos ore. S. H. Dolbear, and Selective Treatment Co., Ltd. (E.P. 254,796, 8.4.25).

Ammonium sulphate (U.S.P. 1,589,809).—See II.

Calcining alkaline-earth carbonates (E.P. 253,305).—See X.

VIII.—GLASS; CERAMICS.

Dilatometric and thermal study of soda-silica glasses. M. O. Samsoen (Compt. rend., 1926, 183, 285—286).—Measurements carried out on soda-silica glasses by the method of Chevenard show a maximum coefficient of dilatation at 15° for a silica content of 56%. A maximum transformation temperature, between two minima, is observed corresponding to the composition $2SiO_2,Na_2O$, which is analogous to alloys and salt mixtures forming eutectics. This and SiO_2,Na_2O are the only definite compounds existing in the silica-soda system in the vitreous state, the minima corresponding to a mixture of these two compounds, containing about 56% SiO_2, and a mixture of silica and the compound $2SiO_2,Na_2O$, containing about 73% SiO_2. Glasses of composition falling on the branch of the curve extending from SiO_2,Na_2O to the first minimum are very easily devitrified. J. Grant.

Patents.

Preparing material for ceramic articles. A. O. Austin, Assr. to Ohio Brass Co. (U.S.P. 1,589,617, 22.6.26. Appl., 1.3.23).—The ingredients are powdered and lawned dry, after which sufficient water is added to produce a mix suitable for forming. This is placed under reduced pressure until the water has permeated the mass. C. Irwin.

Annealing glass. V. F. Feeny. From Illinois Pacific Glass Co. (E.P. 254,580, 30.10.25).—See U.S.P. 1,585,542; B., 1926, 585.

Manufacture of plastic material out of non-plastic oxides. O. Ruff (U.S.P. 1,591,563, 6.7.26. Appl., 18.11.22. Conv., 24.11.21).—See G.P. 382,556; B., 1924, 96.

IX.—BUILDING MATERIALS.

Thermal behaviour of puzzuolana and its reaction in the solid state with some alkaline-earth oxides and carbonates. G. Malquori (Gazzetta, 1926, 56, 42—51; cf. A., 1923, ii, 758).—In the heating of puzzuolana two critical points are observed at about 550° and 900—1060° respectively. As in the case of kaolin chemical changes occur at these points. Puzzuolana reacts with barium oxide at the same temperature as does alumina and kaolin and in the same way; it lowers the temperature of dissociation of barium carbonate to the same extent as alumina and kaolin. The behaviour of the alumina in puzzuolana towards solutions of hydrochloric acid and calcium hydroxide shows the same characteristics as that of free alumina and the alumina of kaolin when these substances have all been heated to the same temperature. The author suggests that the results indicate the existence of free silica and alumina as the principal constituents of puzzuolana, as a silicate of aluminium would scarcely behave in this way. This would explain the setting of puzzuolana, and in view of the differences in the behaviour of silica and alumina according to their origin and treatment, it would also explain the variation in the mortars formed by treatment with lime. S. B. Tallantyre.

Determination of uncombined lime in Portland cement. W. Lerch and R. H. Bogue (Ind. Eng. Chem., 1926, 18, 739—743).—Portland cement contains compounds which are readily decomposed by water or weak acids so that any method involving the use of aqueous solutions is unreliable. The following modification of Emley's method is therefore recommended: 1 g. of finely ground cement is heated to boiling with 25—30 c.c. of absolute alcohol and 5—6 c.c. of anhydrous glycerol, and the mixture is titrated with a $0{\cdot}2N$ solution of ammonium acetate in absolute alcohol using phenolphthalein as indicator. Boiling and titrating are continued until the pink colour does not reappear on boiling for several minutes. The solution is standardised against 0·1 g. of pure lime made by heating calcite to 900° in a platinum crucible. Alkalis and magnesia present in cement do not affect the results. A. R. Powell.

Destruction of cement drain-pipes in soil. K. Nehring (Z. angew. Chem., 1926, 39, 883—887).—Experiments are described which indicate that when cement hardens in air carbon dioxide first combines with the calcium hydroxide after which the calcium aluminate and then the silicate are decomposed; the silica thus liberated is converted into the insoluble form. When the calcium carbonate in the cement exceeds 75% the pipes are much more easily damaged. From a detailed examination of two specimens of corroded drain-pipe and the soil from which they were removed (sandy loam) it is concluded that the free carbon dioxide in the groundwater is responsible for the destruction. The calcium compounds in the cement are first converted into carbonate and then dissolved out in the form of bicarbonate. Magnesium compounds, also, may sometimes take part. S. K. Tweedy.

Patents.

Process of treating slurry. R. C. Newhouse, Assr. to Allis-Chalmers Manuf. Co. (U.S.P. 1,589,712, 22.6.26. Appl., 30.1.25).—Slurry-forming material is finely ground in presence of water, and the heated slurry so formed is cooled sufficiently rapidly to prevent the absorption of enough water to cause setting. A. Cousen.

Impregnated woods and processes of treating woods. J. R. Coolidge (E.P. 254,795, 8.4.25).—See U.S.P. 1,556,570; B., 1925, 960.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Formation of hair copper in copper mattes. System copper–iron–sulphur. H. TIEDEMANN (Metall u. Erz, 1926, 23, 200—210 ; Chem. Zentr., 1926, I., 3624—3625).—The constitution of the system copper sulphide–ferrous sulphide–sulphur has been examined by microscopical, thermal, and conductivity methods. The composition of all technical copper matters lies on a line joining Cu_2S with Fe_2S_3. Mixtures of cuprous and ferrous sulphides during cooling from 584° to 180° slowly decompose with the precipitation of hair copper and the formation of bornite ($3CuS_2, Fe_2S_3$). The deposition of copper is at a maximum in fused mixtures containing 62% Cu. These facts probably explain the occurrence of native hair copper in deposits of bornite and copper pyrites. A. R. POWELL.

Sand-cast alloys of aluminium containing cobalt. S. DANIELS (Ind. Eng. Chem., 1926, 18, 686—691).—Addition of more than 0·5% Co to aluminium or to any of the common aluminium alloys increases considerably the shrinkage on solidification and decreases the tensile strength and ductility. The structure becomes more coarsely crystalline and the castings fuller of blow-holes as the cobalt content and, therefore, the casting temperatures are increased. The eutectic of aluminium and $Al_{13}Co_3$ contains about 6% Co and melts at 644° ; it usually appears as spherulitic or acicular bluish-grey aggregates in the microsection and its character remains unchanged even after prolonged annealing. Cobalt–aluminium alloys are superior to copper–aluminium alloys in the salt-spray corrosion test. A. R. POWELL.

Analysis of commercial magnesium. BONNARD and DANDURAND (Ann. Chim. analyt., 1926, 8, 197—198).—A claim for priority. The method described by Guérin (B., 1926, 326) was published by the authors in Revue d'Artillerie, 1925, 507. A. R. POWELL.

Deformation of tungsten single crystals. F. S. GOUCHER (Phil. Mag., 1926, [vii.], 2, 289—309 ; cf. B., 1924, 792 ; Phil. Mag., 1924, 48, 800).—The deformation under constant load of individual crystals in pure tungsten wires annealed at 3100° K. has been determined at 2000° and 1000° K. Similar measurements have been made on Pintsch single-crystal wires at lower temperatures. The time–extension curves for tungsten crystals show an initial rapid decrease in rate of flow, corresponding to a rapid increase in internal hardening, followed by a longer period at constant rate, over which hardening is proportional to the increase of stress. The hardening is removed by annealing at 2500° K. for 2 min. An *X*-ray study of the crystals reveals distortions of a crystallographic nature which are roughly proportional in amount to the deformation, and are not altered on annealing. The crystals deformed at 1000° show no difference in resolving power in the hardened state and after annealing, whereas the Pintsch wires deformed at the ordinary temperature show an impaired resolving power which improves on annealing. The experiments support the view that hardening is due to residual elastic strains produced by the deformation process rather than to crystal breakdown. A. B. MANNING.

See also A., Aug., 783, **Changes in crystal orientation of copper during recrystallisation** (TAMMANN and MEYER). 786, **Densities of molten lead–cadmium, cadmium–tin, and zinc–tin alloys and of molten cadmium** (SAUERWALD) ; **Heats of mixing of molten metals** (MAGNUS and MANNHEIMER) ; **Metallographic observations on cuprous oxide in copper** (SIEBE) ; **Miscibility of copper and of tin with iron in the molten state** (RUER and KUSCHMANN) ; **Supersaturated mixed crystals and nature of martensite** (RUER). 799, **Ternary system iron–chromium–carbon** (VON VEGESACK). 805, **Rapid corrosion of metals by acids within capillaries** (EVANS).

PATENTS.

Utilising low-grade iron ore. H. H. HINDSHAW, Assr. to HINDSHAW ENGINEERING & DEVELOPMENT Co. (U.S.P. 1,588,420, 15.6.26. Appl., 12.12.24).—The crushed ore is converted into magnetic oxide by heating at 300—600° with a solid carbonaceous material in a limited supply of air. L. M. CLARK.

Process and apparatus for melting iron. Y. A. DYER (U.S.P. 1,589,589, 22.6.26. Appl., 4.2.25).—A deep bed of carbonaceous material is ignited in presence of a supply of air and of the metal to be melted. In a second combustion chamber, the hot waste gases are passed under and through a thin layer of carbonaceous material in presence of additional metal for fusing. L. M. CLARK.

Metal compositions. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of N. H. ADAMS (E.P. 245,437, 19.12.25. Conv., 31.12.24).—See U.S.P. 1,552,184 ; B., 1925, 855. A material suitable for resistance welding electrodes and for bearings is prepared by compressing comminuted tungsten into the desired form, heating at 1250—1350°, and introducing into contact with the sintered mass, in an atmosphere of hydrogen, copper heated above its melting point.

Roasting or calcining ores consisting substantially of alkaline-earth carbonates or of iron carbonate. A. APOLD and H. FLEISSNER (E.P. 253,305, 18.12.24).—Ferrous carbonate or alkaline-earth carbonates are calcined in a shaft furnace by means of a current of hot air, with or without steam, the rate of passage of the hot gases being so regulated that the carbon dioxide evolved is rapidly removed from the furnace. In this way a lower temperature can be used, the time required is shortened, and a considerable economy of fuel is effected. A. R. POWELL.

Concentration of ores. S. Tucker and Minerals Separation, Ltd. (E.P. 253,587, 16.3.25).—Complex ores, the constituents of which fall into four specified groups including (i) galena, compound sulphides of lead with antimony, bismuth, and silver, and copper sulphides such as chalcopyrite, (ii) zinc blende, (iii) iron pyrites, and (iv) tin-containing material such as cassiterite and franckeite, are successively separated in this order by froth-flotation. The ore is washed for removal of soluble constituents. Removal of the first group is effected in a neutral or alkaline pulp by use of a limited quantity of frothing agent. Alkalinity is attained by addition of caustic soda. Addition of copper sulphate to the alkaline pulp, followed by that of a frothing agent such as coal-tar oil or blast-furnace creosote suffices for removal of the second group. The third group is removed by slight acidification of the circuit-liquor and repeated froth-flotation. Gangue is then removed from the cassiterite by gravity-concentration.
L. M. Clark.

[Ductile] alloy. T. H. Reighard (U.S.P. 1,588,468, 15.6.26. Appl., 24.10.22).—A flux manufactured from furnace slag is added to molten copper. The requisite proportion of aluminium is added and an arc heater introduced into the molten mass. After considerably raising the temperature in this way the alloy is allowed to cool. S. Pexton.

Alloy of tantalum. P. H. Brace, Assr. to Westinghouse Electric and Manuf. Co. (U.S.P. 1,588,518, 15.6.26. Appl., 18.4.19).—An alloy resistant to acids and oxidation contains 5 to 30% of chromium, the remainder being tantalum and nickel in the proportions of 1 to 75% of nickel and 25 to 99% of tantalum. L. M. Clark.

Metallurgical process. R. D. Pike and G. H. West (U.S.P. 1,588,806, 15.6.26. Appl., 8.7.24).—Ores, *e.g.*, copper concentrates, are digested with ferric chloride solution to dissolve all the soluble metals and the solution is treated with ferrous sulphide sufficient to reduce the ferric salt to ferrous and to precipitate certain metals at temperatures above 100°. A. R. Powell.

Method of ore treatment. J. T. Terry, jun., Assr. to Metals Recovery Co. (U.S.P. 1,589,615, 22.6.26. Appl., 19.10.14).—Certain metals are dissolved from an oxidised ore by a suitable solvent and reprecipitated in the pulp in elementary form. The pulp is then concentrated by settling.
C. Irwin.

Chromium plating. K. W. Schwartz, Assr. to Chromium Products Corp. (U.S.P. 1,589,988, 22.6.26. Appl., 11.11.24).—Metallic chromium anodes are used in acid plating baths containing chromic acid as the predominating acid. A. R. Powell.

Coating iron and iron alloys with chromium. S. Kyropoulos (U.S.P. 1,590,101, 22.6.26. Appl., 23.3.25. Conv., 26.7.23).—A superficial zone of iron containing a small percentage of carbon is prepared on the article, which is then coated with chromium and tempered. L. M. Clark.

Plating with chromium. J. M. Hosdowich, Assr. to Chromium Products Corp. (U.S.P. 1,590,170, 22.6.26. Appl., 10.9.25).—To improve the throwing power of chromium baths containing the metal in two different states of oxidation, a small quantity of a soluble salt of a metal more electronegative than chromium is added to the bath.
A. R. Powell.

Preventing the adherence of zinc oxide crusts to the walls of the condenser. Urbasch & Co. (G.P. 427,827, 5.12.24).—The interior surface of zinc condensers is coated with a mixture of a basic self-binding material (cement) and a non-binding material containing alkaline-earth oxides or magnesia with or without the addition of a rich loam. The mixture must contain a certain definite proportion of lime or it is useless. A. R. Powell.

Purification of copper. Ges. für hüttenmännische Verfahren m.b.H. (G.P. 427,915, 24.5.24).—Copper cathodes, scrap or alloys, or black copper are melted in an oxidising atmosphere to remove impurities and subsequently deoxidised by the addition of zinc and lead sufficient to combine with the oxygen content of the copper. Finally the metal is melted *in vacuo* to remove excess of zinc. If desired the zinc may be added in the form of brass. A. R. Powell.

Purification of mercury by distillation. Siemens & Halske A.-G. (G.P. 428,023, 2.8.24).—Mercury is heated under pressure in a chamber separated from a second chamber under reduced pressure by a porous partition which is permeable only to mercury vapour. A. R. Powell.

Manufacture of pig iron. C. Davies, jun. (E.P. 254,442, 27.4.25).—See U.S.P. 1,535,109; B., 1925, 596.

Reduction of alumina. Metal Research Corp. (E.P. 245,421, 13.11.25. Conv., 30.12.24).—See U.S.P. 1,551,615; B., 1925, 854.

Desulphuration of zinc ores. W. J. Mellersh-Jackson. From Soc. Anon. des Mines et Fonderies de Zinc de la Vieille-Montagne (E.P. 254,389, 1.4.25).—See G.P. 401,599; B., 1925, 14.

Reduction of ores with continuous regeneration and transformation of the residual gas produced during reduction into fresh reducing gas. G. Constant and A. Bruzac (U.S.P. 1,591,470, 6.7.26. Appl., 7.8.23. Conv., 22.8.22).—See E.P. 202,970; B., 1925, 13.

Producing magnesium-silicon alloy. I.-G. Farbenind. A.-G., Assees. of W. Schmidt (U.S.P. 1,592,160, 13.7.26. Appl., 15.11.23. Conv., 17.11.22).—See G.P. 397,346; B., 1924, 876.

Apparatus [tuyère] for the introduction into furnaces and blast furnaces of materials or residues. G. de Bethune (E.P. 254,396, 3.4.25).

Apparatus for testing the hardness of materials [metals]. T. Hough, and J. Tickle & Co., Ltd. (E.P. 254,465, 14.5.25).

Reclaiming spent pickling solutions (U.S.P. 1,589,610).—See VII.

XI.—ELECTROTECHNICS.

Electrolysis of sodium chromate with a mercury cathode. I. Stscherbakov and O. Essin (Z. Elektrochem., 1926, **32**, 396—399; cf. B., 1925, 710).—In the electrolytic production of dichromate from chromate a sudden rise in the conductivity of the electrolyte is observed when the composition corresponds to the polychromate, $Na_2Cr_4O_{13}$. In order to obtain the best yields of dichromate, electrolysis may be conducted either in normal chromate solutions at high current density or at lower current density in solutions of the above polychromate composition. W. A. Caspari.

See also A., Aug., 801, **Electrolysis of molten sodium-mercury alloys** (Kremann, Krieghammer, and Gruber-Rehenburg); **Electrolysis of molten bismuth-tin alloys** (Kremann, Krieghammer, and Tröster); **Electrolysis of molten tin-cadmium alloys** (Kremann and Baukovac). 802, **Electrolysis of molten alloys of aluminium with magnesium, antimony, zinc, and silver** (Kremann and Dellacher); **Electrolysis of molten metal sulphides and phosphides** (Kremann and Baukovac); **Electrolysis of molten alloys of silver with tin, antimony, bismuth, and lead** (Kremann and Bayer). 804, **Cathodic deposition of metals** (Fröhlich, Clark, and Aborn).

Patents.

Transformer and switch oils. I.-G. Farbenind. A.-G., Assees. of C. Thode and A. Benischek (G.P. 427,744, 12.4.25).—The use of liquid triaryl phosphates is claimed. L. A. Coles.

[Electric induction] apparatus for the treatment of gases at high temperatures. M. Fourment (E.P. 244,749, 7.12.25. Conv., 22.12.24).—See F.P. 593,298; B., 1926, 145.

Leading-in means, particularly for high-tension currents [in electrical gas purifying apparatus]. Lodge-Cottrell, Ltd. From Metallbank & Metallurgische Ges. (E.P. 254,605, 29.12.25).

Apparatus for refining oil (U.S.P. 1,586,132)—See II.

Treating hydrocarbons and oils (U.S.P. 1,588,308).—See II.

Plating with chromium (U.S.P. 1,589,988 and 1,590,170).—See X.

Preservation of sap-containing feeding-stuffs (Swiss P. 106,942).—See XIX.

XII.—FATS; OILS; WAXES.

Determination of the titer of tallow. J. Davidsohn (Z. Deuts. Oel- u. Fett-Ind., 1926, **46**, 353—354).—The effect of varying the conditions in the Dalican method of determining the titer (solidif. point of insoluble fatty acids) of tallow was determined. A particular tallow gave the following results: (*a*) without stirring, titer 43·50° and 43·40°; (*b*) stirring three times to the right and three times to the left after solidification started, 43·48° and 43·40°; (*c*) stirring from the start until solidification commenced, 44·20°. It is recommended to omit the use of alcoholic potash for the saponification, which can be effected by heating the fat and aqueous alkali on a water bath, and then keeping the mixture at 100—105° for 1½ hrs. This is much safer than the alcoholic alkali saponification, as there is no risk of ester formation from traces of alcohol left in the mixture. In the case of the above tallow, when the alcohol was not completely removed after saponification with alcoholic alkali a titer of (*a*) 39·90°; (*b*) 40·82°, and (*c*) 40·75° under the conditions mentioned was observed. It is stated that the latent heat of fusion of the fatty acids decreases with repeated reheating and finally neither rise nor stationary point is observed in the test; in such cases a fresh sample must be taken. A. Rayner.

New fatty acids in shark liver oil. Fatty acids of shark and ray liver oils. I. M. Tsujimoto (Z. Deuts. Oel- u. Fett-Ind., 1926, **46**, 385—388).—The fatty acids from two species of red shark were examined, and two new fatty acids were isolated, viz., *selachoceric acid*, $C_{24}H_{48}O_2$, white crystals, m.p. 78°, and *selacholeic acid*, $C_{24}H_{46}O_2$, white crystals, m.p. 42·5—43°. The latter when hydrogenated gave a saturated acid, iso*selachoceric acid*, m.p. about 82°. By treating selacholeic acid with nitrous acid, a stereoisomeride, *selachelaidic acid*, m.p. 60·5°, was produced. A. Rayner.

Test for Chinese wood [tung] oil. E. R. Bolton and K. A. Williams (Analyst, 1926, **51**, 335—338).—Adulteration of tung oil by small proportions (5%) of other oils may be detected and information as to the nature of the adulterant obtained by applying a modification of Worstall's test. 150 g. of oil are heated in an aluminium beaker of 3 in. diameter, and approximately 4 in. high, so as to reach 285° in 4 min., the mixture being vigorously stirred by the thermometer. At 285° a stopwatch is started and the time noted to the point where the oil fails to drop, the temperature meanwhile being kept constant. Genuine oils reach this point in less than 8 min. 2 g. are then cut from the centre of the resulting polymerised mass, incorporated with about 3 g. of silver sand and 2 c.c. of light petroleum, and extracted with light petroleum. Pure tung oils give an extract of 28% with a variation not exceeding 2% on either side. The percentage of foreign oil is found by deducting 30·0 from the extract per cent., and multiplying by 2·70 if the adulterant is linseed oil or other oil of similar high iodine value, and by 1·33 for other oils. D. G. Hewer.

Polymerisation of linseed and sunflower oils in the form of their soaps. G. S. Petrov and S. J. Dimakov (Z. Deuts. Oel- u. Fett-Ind., 1926,

46, 417—418).—In the oxidation or polymerisation of glycerides, it is known that loss of glycerol occurs, acraldehyde being almost invariably formed. This fact was quantitatively confirmed by the authors, who oxidised two samples of linseed oil at 180° for 20 and 50 hrs. respectively, the sp. gr. rising to 0·950 and 0·989 while the glycerol content fell from the theoretical 10·5% to 8·76% and 5·95%. The desirability of polymerising the soaps of drying and semi-drying oils rather than the original glycerides was therefore suggested. Fatty acids were isolated from soaps of linseed and sunflower oils that had been heated under pressure for 5 hrs. at 160—200°, alone and in the presence of various amounts of alkalis. The presence of free alkali during the treatment caused a marked increase in the viscosity and decrease in the iodine value of the acids, while in the absence of much free alkali, the polymerisation proceeded best when the soap was introduced directly into the autoclave (no special containing vessel being used). S. S. WOOLF.

Inhibiting agents in the oxidation of unsaturated organic compounds. O. M. SMITH and R. E. WOOD (Ind. Eng. Chem., 1926, 18, 691—694).—By measurement of the volume of oxygen absorbed the authors have examined the effect of numerous substances on the atmospheric oxidation of oils and fats, fatty acids, soaps, and rubber. Active inhibitants may be divided into two main groups, powerful reducing agents such as stannous compounds, sodium thiosulphate, etc., and strong bases, particularly amines. The action of sodium hydroxide, carbonate, silicate, and phosphate is possibly due to their combination with substances produced by oxidation which themselves act as catalysts. Aromatic alcohols and phenols are also effective as inhibitants. The inhibiting substance will prevent oxidation at any period during the oxidation and in general stability and resistance towards oxidation increase with the concentration of the inhibitant. In some cases, *e.g.*, phenols, such as eugenol, there is a concentration at which the inhibitant is most effective and above which it acts as a positive oxygen catalyst. The influence of the inhibitant is only temporary, the period depending on the temperature, concentration, and products of oxidation. After the effect of the anti-oxidant is lost the rate of oxidation is the same as before. It is suggested that the basic inhibitors combine with acidic oxidation products and prevent them acting as autocatalysts in the oxidation, or that the partial valencies of the tervalent nitrogen atom (or other elements with free valencies) form intermediate compounds with the easily oxidised ethylenic carbon atoms. This temporary compound controls the rate of oxidation for a definite but limited period of time. R. BRIGHTMAN.

Myricyl alcohol [from carnauba wax]. S. GOTTFRIED and F. ULZER (Chem. Umschau, 1926, 33, 141—145).—Myricyl alcohol was isolated from carnauba wax as follows. The wax was purified by the removal of 3—5% of impurities by extraction with alcohol in a Soxhlet apparatus. It was then saponified with alcoholic potash for 48 hrs., the alcohol was removed by evaporation, the soap and free alcohols were dissolved in hot water, and the excess alkali was removed by successive solution and precipitation of the soaps with salt. From this product the unsaponifiable matter was extracted by means of trichloroethylene, which is a better solvent than light petroleum. The unsaponifiable matter was next acetylated by acetic anhydride, and the neutral esters were separated by a series of fractional distillations. The following fractions were separated at 10 mm. pressure: (*a*) b.p. 268—270°, m.p. 58·5—59°, identified after recrystallisation as *n*-heptacosane; (*b*) b.p. 296—298°, m.p. 62—62·5°; yielded by fractional precipitation from acetone and chloroform solution and subsequent saponification ceryl alcohol; (*c*) b.p. 312—314°, by one crystallisation from a mixture of one part of trichloroethylene and 5 parts of alcohol gave an acetate, which on saponification yielded an alcohol shown to be myricyl alcohol and amounting to 45% of the raw carnauba wax. The myricyl alcohol had the composition $C_{30}H_{62}O$, not $C_{31}H_{64}O$ as stated by Gascard (B., 1924, 140). A. RAYNER.

Determination of clouding point of soap solutions. K. BRAUN (Z. Deuts. Oel- u. Fett-Ind., 1926, 46, 370; cf. B., 1926, 135).—The advantages of making the determination on a solution having a definite concentration of fatty acids rather than of soap are emphasised, but a 1% solution could be used equally as well as the 3% solution previously suggested. A. RAYNER.

PATENTS.

Separating oil from solid vegetable substances. K. HOLTER and S. THUNE (E.P. 246,527, 15.4.25. Conv., 13.9.24. Addn. to 232,601; B., 1926, 448).—The process for treating blubber etc. described in the chief patent may be applied in the treatment of oil-bearing vegetable substances. A. RAYNER.

Apparatus for distilling fatty acids. H. BOLLMANN (E.P. 246,803, 18.11.25. Conv., 2.2.25).—Distillation is effected under vacuum in a column still, preferably constructed of firebrick and surrounded by a metal jacket. The fatty acids enter at the top of the still and pass downwards over a retarding filling, *e.g.*, Raschig rings, supported on a series of partitions fixed to the side of the still, the partitions having central openings to facilitate the passage of the fatty acids and of the steam, which enters at the bottom of the still. A. RAYNER.

Purification of oils and fats. A. S. KONSTAS, and SOC. ANON. IND. DES MATIÈRES GRASSES ET SAVONS "VELOS" (E.P. 252,949, 8.9.25).—To recover oil carried down in the soap stock in the alkali refining of oils, the mixture is extracted with a solvent for the soap which does not appreciably dissolve the oil, and is also miscible with water. After separation of the undissolved oil, the solvent, *e.g.*, methylated spirit, is recovered from the soap

solution by circulating the latter in contact with a boiling solution of common salt, contained in a vessel divided by a partition into two compartments, communicating with one another through apertures in the lower part of the partition. The soap solution is heated, so that ebullition takes place only in one compartment, the precipitated soap being carried to the surface of the liquid in the other compartment free from the volatile solvent, which is distilled over with the steam and recovered. A. Rayner.

Refining oils and fats. F. C. Gephart, Assr. to Cocoa Products Co. of America (U.S.P. 1,588,405, 15.6.26. Appl., 11.5.23).—Oils and fats are refined by heating with willow charcoal and infusorial earth. A. Rayner.

Refining. [Deodorising oils.] H. M. Lamy-Torrilhon (U.S.P. 1,592,112, 13.7.26. Appl., 10.4.24. Conv., 14.4.23).—See E.P. 214,273; B., 1926, 99.

Lubricating oil (U.S.P. 1,587,859).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Glycol ethers and their use in the lacquer industry. J. G. Davidson (Ind. Eng. Chem., 1926, **18**, 669—675).—The solubility of cellulose nitrate in alcohol–ether mixtures suggested the probable utility of alkyl ethers of the alkylene glycols as "two-type" solvents for cellulose lacquers. A large number of glycol and polyglycol ethers showed excellent solvent properties and pronounced superiority over the ester type of cellulose ester solvent, the monoethyl ether of ethylene glycol being particularly suitable. It has b.p. 134·8°/743 mm., d_{15}^{15} 0·9360, n_D^{24} 1·4042; it mixes in all proportions with most organic solvents, and forms with water a mixture of constant b.p. (98—99°), containing 40% of the ether by volume. Its dilution ratio (proportion of non-solvent that causes precipitation of cellulose nitrate from a 10% solution in the solvent) is unusual, ranking higher than that of ethyl lactate and much higher than that of butyl acetate for the three diluents toluene, light petroleum, and water. Its powerful, but slow, solvent effect permits of the retention of considerably more than the usual amounts of cellulose nitrate, and therefore pigment, in a lacquer of consistency suitable for brushing, while the need for plasticisers is obviated or lessened. Lacquers made with this solvent thus dry harder and more resistant to abrasion than the normal, whilst they have greater hiding power and may be brushed out without affecting the previous coats. They are free from residual odour and do not deteriorate on storage by the development of acidity, hydrolysable ester groups being absent from the solvent. Though this ether is not a universal resin solvent, the difficultly soluble gum dammar and ester gum may, with some preliminary treatment, be rendered compatible with cellulose nitrate in this solvent. S. S. Woolf.

Deposits in varnishes. F. Wilborn (Farben-Ztg., 1926, **31**, 2351).—To investigate the causes of clouding in varnishes, widely attributed to the progressive formation of insoluble lead salts of hydroxy-acids, the author prepared varnishes by incorporating a fixed weight of lead–manganese resinate, in the absence of air, with samples of raw linseed oil and the same oil air-blown for 6, 12, and 24 hrs. respectively at 120°. The iodine values of the four oils were 176·6, 172·5, 168·2, and 163·0, and the relative viscosities 100, 112, 133, and 164 respectively. The drying times of the corresponding varnishes were 8 hrs. 25 min., 8 hrs. 5 min., 7 hrs. 30 min., and 6 hrs. 5 min. The varnishes (which approximated to each other in colour, although the blown oils were appreciably bleached) were observed in glass tubes over a period of 6 months. Contrary to anticipation, there was no heavier sediment in the varnishes made from blown oil than in that from raw oil. S. S. Woolf.

Joint use of cobalt–lead driers. F. Wilborn (Farben-Ztg., 1926, **31**, 2352—2353).—The lack of mechanical strength in films in which cobalt is the sole drier may be compensated by the use of lead drier in conjunction with the cobalt, the extreme rapidity of drying induced by the latter still being evident. The author has observed the drying times of varnishes containing various proportions of these metals added as resinates (containing 6·5% of cobalt and 23·1% of lead respectively). Three composition–drying time curves, corresponding to total resinate contents of 3%, 2%, and 1·5% respectively, are given, the drying time in each case rising slowly from the cobalt resinate alone to the 1 : 1 mixture, further increase in the proportion of lead resinate causing a more rapid rise in drying time. A total of 2% of cobalt–lead resinate is recommended, distributed according to the drying time and strength of film required. S. S. Woolf.

Reactions between solid substances reduced to the colloidal state. Sterkers and Bredeau.—See A., Aug., 792.

Polymerisation of linseed and sunflower oils. Petrov and Dimakov.—See XII.

Patents.

Apparatus for extracting rosin and turpentine from wood. McG. Cline (U.S.P. 1,589,837, 22.6.26. Appl., 25.8.22).—Finely divided wood is progressively forced down through a retort and discharged, being in contact with a counter-current of steam throughout. S. S. Woolf.

Manufacture of artificial resins. A. Regal (E.P. 254,888, 29.7.25).—See U.S.P. 1,584,472; B., 1926, 596.

Nitration of petroleum distillates (U.S.P. 1,588,027).—See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Needle form of the caoutchouc molecule as a practical structure theory. E. Lindmayer (Gummi-Ztg., 1926, 40, 2261—2262).—The hypo-

thesis is put forward that the rubber molecule consists of a chain of which the length is expressed by x in the formula $(C_5H_8)_x$, and is of the order of 14 times the diameter. The properties of raw rubber and the changes induced by physical manipulation and by vulcanisation are considered from this point of view. S. I. LEVY.

Thermodynamics of the Joule effect in raw rubber. L. HOCK and S. BOSTROEM (Kautschuk, 1926, 130—136; cf. B., 1925, 816).—The hysteresis diagrams for a sample of raw smoked sheet rubber on first and second extensions have been constructed from data obtained with a specially modified Schopper machine, and the values compared with those obtained for the same sample after vulcanisation. The hysteresis loops for both raw and vulcanised material for a first elongation of 600% enclose approximately the same areas, but the work done in extension is about four times as great with the latter; the relatively much smaller hysteresis loss for the vulcanised material corresponds with the much smaller permanent extension, and the similarity of the first and second extension curves with this material. The second extension curve for the raw rubber is considered to correspond more closely with the true internal structure of the rubber than the first, which depends on the more or less accidental structure of the coagulum. S. I. LEVY.

Agglomeration density of rubber in relation to aggregation, vulcanisation, deformation, and temperature. M. KRÖGER (Gummi-Ztg., 1926, 40, 2319—2321).—Aggregation, vulcanisation, deformation, and reduction of temperature lead to increase in the density of raw rubber. *X*-Ray examination of test pieces after keeping for five years showed two diffuse rings. No effect on ultraviolet light could be observed with a rubber–sulphur chloride gel. S. I. LEVY.

Heat reactions occurring during vulcanisation of rubber. A. A. PERKS (J.S.C.I., 1926, 45, 142—149T).—Combination of rubber and sulphur is slightly exothermic in the early stages, but after 4—5 pts. of sulphur have combined with 100 pts. of rubber a strongly exothermic reaction sets in, and the temperature may rise 100—150° above the vulcanising temperature, with evolution of hydrogen sulphide. With pale crêpe the maximum effect was observed with a mixing of 79 pts. of rubber and 21 pts. of sulphur. The removal of the resin delays the start of the reaction, but the presence of accelerators hastens it and appears to cause greater evolution of heat. The necessity for long cures at low temperatures for ebonite mixings is thus made clear. Mixings containing over 10% of sulphur show an arrest in the temperature–time curve at 100°, due mainly to changes occurring in the sulphur. S. I. LEVY.

Prevention of lead poisoning in industry. I. Rubber industry. C. A. KLEIN (J. Ind. Hyg., 1926, 8, 296—299).—The method adopted, by which the manufacturer supplies to the rubber goods factories a mixture of 80 pts. of the lead compound with 20 pts. of rubber or wax in the form of thin sheets which do not give rise to dust, has proved very successful in preventing poisoning in the latter factories. S. I. LEVY.

Inhibiting agents in oxidation of unsaturated organic compounds. SMITH and WOOD.—See XII.

PATENTS.

Caoutchouc compositions. A. BIDDLE (E.P. 253,740, 31.7.25).—Waterproof adhesive and glue-like compositions are prepared by incorporating casein and a soluble silicate with rubber, gutta-percha, or balata latex. S. I. LEVY.

Treatment of rubber. F. C. JONES (U.S.P. 1,591,132, 6.7.26. Appl., 5.3.23. Conv., 11.3.22).—See E.P. 199,095; B., 1923, 789 A.

XV.—LEATHER; GLUE.

Alkaline hydrolysis of hide. E. AGENO-VALLA and G. BORNATE (Boll. Uff. Staz. Sperim. Ind. Pelli, 1926, 4, 83—89).—The fact that preliminary treatment with lime renders difficult the unhairing of hide even by concentrated sodium sulphide solution is not due to any specific action of the lime, such as insulation of the fibre by precipitated calcium sulphide. Similar protective action is, indeed, exerted by other alkaline hydroxides in low concentration. The experimental results obtained fail to support Merrill's suggestion (cf. B., 1925, 18, 327) that the hydrogen-ion concentration of the solution is the determining factor; preliminary treatment of the hide with acid appears to aid subsequent hydrolysis of the keratin by sodium sulphide. The presence of hydroxyl ions should diminish the dissociation of the keratin as a base and cause it to function solely as an acid, with formation of the corresponding salt, but this could scarcely result in a protective action. Possibly the hydroxyl ions cause an intramolecular transposition, such as that of the NH·CO group into NC·OH or that of the groups united to the benzene nucleus, and thus produce increased stability. T. H. POPE.

Determination of the proteolytic power of bating materials. L. CHIESA (Boll. Uff. Staz. Sperim. Ind. Pelli, 1926, 4, 112—122).—Determinations of the proteolytic powers of three bating materials by Lepetit's method (B., 1924, 481) and by the casein method give results of the same order in the two cases. As a rapid means of control, the following modification of the casein process is suggested. A casein solution is prepared by precipitating casein by a 1% acid solution in presence of a trace of organic calcium salts and dissolving the precipitate in water containing just enough 10% sodium hydroxide solution to turn the liquid pink on addition of phenolphthalein solution. A 5% aqueous solution of the bating material is prepared at 38—40°; the liquid is shaken occasionally and allowed to settle, the comparatively clear liquid being used. To 20 c.c. quantities of the casein solution in test-tubes are added 2 c.c. of 5% solutions of the different bating materials, the liquids being mixed and the

tubes kept at 38—40° for an hour and then placed in boiling water and left there for 1 min. after a precipitate appears. The value of the bating material is shown by the volume of the precipitate which subsequently settles, and by the clearness of the supernatant liquid. The results obtained may be checked by filtering off the precipitates and precipitating the residual undecomposed casein by addition of 5 c.c. of 10% acetic acid solution. This method gives results in agreement with Lepetit's method. The use of commercial casein may be avoided as follows: to 100 c.c. of fresh milk at 35° are added 10 c.c. of the 5% solution of the bating material; the mixture is vigorously stirred and then left at rest at 35° and the time measured from addition of the material until coagulation occurs; this is the case when a wisp of straw, immersed vertically in the liquid until it touches the bottom of the beaker, remains upright. The most active bating enzyme will cause coagulation in the shortest period of time.

T. H. POPE.

Combining power of various vegetable tanning substances with the protein of hide. C. SCHIAPARELLI and C. AVENATI-BASSI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1926, 4, 134—146).—Previous investigations on the extent and manner of absorption of natural tanning agents during tanning, mostly carried out with hide powder, are briefly reviewed. The experiments now described show that, of the three materials, sulphited quebracho, sumac, and *tannin puriss.* (Kahlbaum), the last gives the greatest, and the first the least, yield; the swelling also is greatest with the pure tannin. Leathers obtained with extracts of oak, quebracho, sumac, chestnut, and myrobalans show diminishing temperature of gelatinisation (cf. B., 1924, 142; 1925, 463, 464) and resistance to hot water in the order given.

T. H. POPE.

[Tanning properties of] complex chromium salts. E. AGENO-VALLA and R. ASTENGO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1926, 4, 69—74).—The results obtained in an examination of the behaviour of a number of complex chromium salts towards hides indicate that the tanning properties of the chromium are peculiar to the chromium ion, which loses these properties when its co-ordination valencies are saturated by various molecules. It seems, therefore, that the secondary valencies play an important part in tanning. An explanation is thus furnished of the difficulty of demonstrating that chrome leather is a true chromium compound and of the fact that part of the acid of the chromium salt remains as an essential constituent of the leather. Some of the salts examined are absorbed by the hide, but exert no tanning action, and others are not absorbed even sufficiently to colour the hide. Those salts having a complex univalent cation are absorbed far more readily than the others; the complex base $[Cr(NH_3)_5H_2O](OH)_3$ is strongly absorbed, probably owing to its alkaline reaction. The hydroxy-salts are usually very slightly soluble and thus not easy to test, but they do not appear to have tanning properties or to be absorbed by hides. If only the ordinary valency of the chromium and that of the anion in the molecule are considered, these salts are basic, the basicity of hydroxydiaquotriamminochromic iodide being 3 : 2 and that of dihydroxydiaquodiamminochromic bromide 3 : 1. Basicity alone is not a sufficient condition for these salts to exert tanning properties; in addition the chromium must be in the ionic state and must have free co-ordination positions.

T. H. POPE.

Determination of chromic oxide in chrome leather. L. CAREGGIO and G. BUSSINO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1926, 4, 123—124).—If, in the ordinary method of determining chromium trioxide in chrome leather, the fusion of the leather with a mixture either of sodium carbonate and potassium nitrate or of sodium carbonate and potassium carbonate and chlorate is effected in a porcelain instead of a platinum crucible, treatment of the mass with hydrochloric acid gives silicic acid, which would interfere with the iodometric titration and must be removed. To this end the liquid is evaporated and the residue dried, both of these operations being carried out on a water-bath. The dry residue is then dissolved in water containing a small quantity of hydrochloric acid, the whole of the silicic acid being thus converted into polysilicic acids, which are insoluble in water or acid and are eliminated by filtration prior to treatment with potassium iodide and titration with iodine.

T. H. POPE.

See also A., Aug., 792, **Concentration and purification of solutions of hydrophilic colloids** (BECHHOLD and HEYMANN). 793, **Swelling and osmotic pressure of gelatin in salt solutions** (NORTHROP and KUNITZ).

PATENT.

Process for brightening dark tannages. FARBW. VORM. MESITER, LUCIUS, & BRÜNING, Assees. of K. DAIMLER (G.P. 423,137, 15.2.22).—The tannage is after-treated with insoluble, light-coloured, non-volatile organic compounds, especially hydroxyaryl compounds, which are less acidic than the tannin which has been used. For instance, leather tanned with the brown tannin prepared from lignite and nitric acid, after previous rinsing, is agitated for 10 min. in a 1—4% solution of β-naphthol in caustic soda or an aqueous solution of the sodium salt of the resin obtained from sulphur chloride and crude cresol. The bleaching liquor is then replaced by 0·25—1% hydrochloric acid and after 5 min. the leather is rinsed in water for 30 min. The colour of the leather is equal to that obtained with oak-tannin and is much brighter than the original. A dark quebracho tannage can be brightened in the same way. In place of the sodium salt of β-naphthol may be used the alkali salts of α-naphthol, chloronaphthols, hydroxyanthracene, cresol–formaldehyde or cresol–paraldehyde condensation products, or alkali-soluble natural resins or lignic acid.

R. BRIGHTMAN.

XVI.—AGRICULTURE.

Suction force of soils. Application to the study of the soil–plant system. J. H. JOFFE and H. C. MCLEAN (Science, 1925, **62**, 548—550).—Since the suction force of a soil is a measure of the colloid content (A., 1925, i., 1372), these data may be correlated with the wilting point of plants. The relation of the phenomenon to irrigation and moisture movement in soils is considered.

A. A. ELDRIDGE.

Separation of the slimy and colloidal components of the soil by centrifuging. J. DUMONT (Compt. rend., 1926, **183**, 131—133).—The use of a centrifuge revolving at 10—150 revs. per sec. is to be preferred to sedimentation processes for the analysis of soils, and produces an increase in the rate of settling of the particles of 52—11,000 times according to their diameters. Samples of crude clay obtained in the physical analysis of soils, have been centrifuged at various speeds in an ammoniacal medium, and may be divided into slime consisting of particles of diameter less than 0·001 mm., and true colloidal clays which remain in suspension. The former vary from 60 to 95% of the total. The method may be made quantitative. J. GRANT.

Effects of lime and potash fertilisers on certain muck soils. F. LOEHWING (Bot. Gaz., 1925, 80, 390—409).—The use of calcium carbonate as fertiliser depressed the potassium content and the potassium : calcium ratio in crops grown on acid muck soils. Application of lime and potash depressed the organic nitrogen and carbohydrate content. High nitrate-nitrogen is associated with high calcium content of tissues and low crop-yield; high crop-yield is associated with high organic nitrogen and high total carbohydrate content.

A. A. ELDRIDGE.

Disinfection of seeds. A. NIETHAMMER (Biochem. Z., 1926, **172**, 173—211).—The disinfection of seeds with bleaching powder is discussed. The nature and strength of the infection, origin, and surface characteristics play an important part in the success or failure of disinfection; *e.g.*, round smooth seeds are easiest to disinfect. Owing to these difficulties the seeds should be gathered under conditions as nearly aseptic as possible.

H. I. COOMBS.

PATENTS.

Manufacture of fertilisers. E. L. PEASE and D. TYRER (E.P. 253,572, 19.2.25).—Finely-ground rock phosphate is treated with sulphuric acid and the mixture is filtered and washed until strong phosphoric acid ceases to be obtained. The calcium sulphate, which still contains phosphoric acid and generally some calcium phosphate, is treated with ammonia and carbon dioxide (or with a solution of ammonium carbonate) until carbon dioxide is no longer absorbed. Calcium carbonate containing phosphate, and ammonium sulphate are thus formed; the former is filtered off, washed, and dried and the latter is recovered by crystallisation from the washings. One quantity of sulphuric acid, used in this manner, thus serves to fix much more than its equivalent of ammonia. C. T. GIMINGHAM.

Manufacture of fertilisers. E. L. PEASE (E.P. 253,580, 16.3 and 7.7.25).—A fertiliser is prepared by mixing ground mineral or organic calcium phosphate with a porous material, such as ground peat, and treating the mixture either with sulphuric acid and water, or, after moistening, with gases containing sulphur dioxide and air or oxygen, or with gases containing sulphur trioxide, and, finally, using the calcium sulphate and phosphoric acid so produced as an absorbent for ammonia.

C. T. GIMINGHAM.

Process of treating phosphate material. H. BLUMENBERG, JUN., Assr. to STOCKHOLDERS' SYND. (U.S.P. 1,588,651, 15.6.26. Appl., 7.8.25).—Ground phosphate rock is mixed with sodium nitrate, ammonium sulphate, and water, and the mixture is treated with sulphur dioxide. Sodium sulphate, ammonium phosphate, ammonium sulphite, and calcium nitrate are thereby formed.

C. T. GIMINGHAM.

Insecticide. E. H. SIEGLER and C. H. POPENOE (U.S.P. 1,589,866, 22.6.26. Appl., 17.11.24).—Lauric acid, or any free aliphatic acid having more than four carbon atoms in the radical attached to the carboxyl group, is claimed as an insecticide for plants. C. T. GIMINGHAM.

XVII.—SUGARS; STARCHES; GUMS.

Electrical conductivity method of determining the ash content of raw cane sugars. F. W. ZERBAN and J. MULL (Facts about Sugar, 1926, **21**, 278).—Using the apparatus described by Tödt (B., 1925, 775), the average values for content of water-soluble ash in 86 raw cane sugars were found to correspond closely with those by incineration, the individual results agreeing within 0·01% in 57% of the samples examined, and within 0·02% in 80%. In only 8 sugars was the discrepancy 0·04—0·06%, and in these probably the error is on the part of the chemical method, or is due to the samples not being uniform. J. P. OGILVIE.

Determination of the hydrogen-ion concentration in the cane sugar industry. L. BAISSAC (Int. Sugar J., 1926, **28**, 370—373).—In Mauritius using the sulphitation method, the optimum p_H after adding lime is very near to 7·0 (cf. Farnell, B., 1925, 416), and at p_H 8·5 (phenolphthalein alkalinity) settling is slower. In the control of the Bach syrup sulphitation process, good filtration was obtained at p_H 6·8—6·9. J. P. OGILVIE.

Separation of the amyloses in some common starches. T. C. TAYLOR and H. A. IDDLES (Ind. Eng. Chem., 1926, **18**, 713—717).—By ultra-filtration through a collodion membrane, prepared by Nelson and Morgan's method (B., 1924, 235), and by electro-dialysis, a clean separation of the slimy, less soluble α-amylose from the mobile, easily dispersed,

β-amylose, in gelatinised corn (maize), rice, and potato starches has been effected. The yields by the two methods are concordant and the β-amylose is obtained in concentrations up to 8%. α-Amylose is only slowly hydrolysed by 10% hydrochloric acid, yielding on complete disintegration a flocculent residue similar to that resulting from the original starch. β-Amylose is readily hydrolysed to a clear colourless solution, with the characteristic properties of a reducing sugar, and the reaction is proposed as a criterion of purity for β-amylose, together with its α_D^{25} 186°, its clear deep blue colour with iodine, its solubility, absence of reducing properties before hydrolysis, and absence of ash. In the case of maize starch the fatty acids are found almost quantitatively with the α-amylose, whilst with potato starch, which contains appreciable amounts of phosphorus, fatty acids are found in both amyloses. The starch used was purified by Taylor and Nelson's method (B., 1920, 668 A) and gelatinised with ammonium thiocyanate, the progress of gelatinisation being followed with the microscope. R. BRIGHTMAN.

Maize and potato starches for textile finishing. EKHARD.—See VI.

PATENTS.

Extraction process and apparatus. H. F. HOLZHEUER (U.S.P. 1,587,646, 8.6.26. Appl., 28.9.25).—In an extraction process where a number of extraction units are arranged in series, as in the beet sugar industry, the pressure of the extracting liquid falls off to such an extent that the process becomes uneconomically slow. The pressure cannot be raised above a certain value at the beginning, or else the first few units would choke, so the liquid is supplied at the greatest economical pressure to the first cell and later removed from the circuit and its pressure raised by means of a "booster" pump, whence it is returned to the next unit of the series. E. S. KREIS.

Refining sugar juice by chlorination. S. OCHI and F. KOTERA (U.S.P. 1,591,879, 6.7.26. Appl., 3.12.24).—See E.P. 249,191; B., 1926, 560.

XVIII.—FERMENTATION INDUSTRIES.

Assimilability of the nitrogen in the nutrient solution by yeast in the aeration process. H. CLAASSEN (Z. angew. Chem., 1926, 39, 880—883; cf. A., 1926, 641).—Experiments are described on the assimilation by yeast of the nitrogen compounds in different molasses, from which it is concluded that beet-sugar molasses is a suitable raw material for the cultivation of baker's yeast. After addition of phosphoric acid better yields of good quality yeast are obtained from beet molasses than from any other raw material. This result is supported by a large-scale experiment. S. K. TWEEDY.

Enzyme purification by electrodialysis and electro-osmosis. FRICKE, FISCHER, and BORCHERS.—See A., Aug., 791.

PATENT.

Yeast food product (U.S.P. 1,589,853).—See XIX.

XIX.—FOODS.

Effect of drying on the vitamins of milk. H. JEPHCOTT and A. L. BACHARACH (Le Lait, 1926, 6, 249—259; Chem. Zentr., 1926, I., 3510).—Vitamins in milk are not destroyed by the usual drying processes provided sound, wholesome milk is utilised. The most satisfactory drying processes are those which prevent oxidation and destruction of the vitamin-*C* and minimise bacterial infection by the exclusion of air. B. W. CLARKE.

Use of commercial rennet in the manufacture of cheese from whey. G. GUITTONNEAU, J. KEILLING, and A. BARRET (Le Lait, 1926, **6**, 170—180, 259—268; Chem. Zentr., 1926, I., 3511).—Excellent cheese can in general be made from whey by the use of commerical brands of rennet. The fundamental biochemical changes underlying the process and details of actual manufacture are described. B. W. CLARKE.

Effect of lactic acid-producing *Streptococci* on the flavour of cheddar cheese. G. J. HUCKER and J. C. MARQUARDT (New York Agr. Exp. Stat. Tech. Bull., 117, Mar., 1926. 13 pp.).—*Streptococcus paracitrovorus*, Hammer, has a favourable influence on the production of the flavour of cheddar cheese prepared from raw or pasteurised milk. *S. citrovorus*, Hammer, has no effect on the flavour, whilst *S. lactis*, Löhnis, has about the same effect as commercial starters (? *S. cremoris*, Orla-Jensen). High-grade milk to which 0·23—0·24% of pure lactic acid is added produces cheese very similar to that obtained by the use of commerical starters. Certain strains of acid proteolytic cocci produce an unpleasant flavour in the cheese, which rapidly becomes soft. L. F. HEWITT.

Vitamin content of foodstuffs. W. H. EDDY (Amer. J. Pub. Health, 1926, **16**, 109—115).—The determination of *A*-, *B*- and *C*-vitamin values of foodstuffs is discussed. Investigation of the value of the banana as an antiscorbutic (vitamin-*C* value) showed the protective dose to be 5 g. per guinea-pig per day. Animals fed on the same diet, believed to contain all the dietary requirements except vitamin-*C*, with addition of 8—10 g. of banana, grew much better than when only the protective amount of the latter was added. The banana was also found to be rich in vitamin-*A*, being a better source of this vitamin than lettuce and, on a dry basis, having 1/5 the value of butter in this respect. Data on green peas and cabbage are also included. R. E. THOMPSON.

Vitamins in heat-sterilised food. C. M. DUGDALE and R. J. MUNRO (J.S.C.I., 1926, **45**, 135—140T).—Finely minced mixtures of various meats of average water content 65—72% and fat 6—10% were packed in 85-g. pots, and these were

heated for a total period of 40 min. at 110°. The centre of the pot is thus kept for about 10 min. at 110°, and at 105—110° for an average of 30 min. Soups were packed in 10-oz. cans and given a total heating of one hour at 115°. These temperatures are adequate for destruction of all harmful bacteria. The foods were fed to rats which had started to lose weight on basal vitamin-free diets. The minimum quantity of minced meats sufficient to supply the daily vitamin-*A* necessary for a rat was found to be about 3 g. and of soups less than 10 g., and for vitamin-*B*, about 7 g. of meat and 5—10 g. of soup. Vitamin-*A* was found to be adequately supplied by 3 c.c. of milk, but for vitamin-*B* more than 15 c.c. were necessary, whilst 2 g. of cabbage were needed for vitamin-*A*, and over 4 g. for vitamin-*B*. D. G. Hewer.

Detection of apple pulp in jam. W. Partridge (Analyst, 1926, 51, 346).—10 g. of jam are mixed with approximately 100 c.c. of warm water so as not to break up any whole fruit, the liquor is strained through gauze of about 20-mesh, preferably into a centrifuge tube, or into a urine sedimenting cylinder, and centrifuged for 1 min. or left to settle for 2 hrs. The deposit thus obtained is much richer in apple cells than was the original pulp. Any iodine-reacting character possessed by the apple cells at the time of manufacture persists for a long time in the jam, *e.g.*, 39 months. D. G. Hewer.

Determination of copper in foodstuffs. L. H. Lampitt, E. B. Hughes, P. Bilham, and C. H. F. Fuller (Analyst, 1926, 51, 327—335).—A rapid method, suitable for routine sorting tests and for a range of copper from 0·07 to 0·40 mg., consists in comparing the colour formed on addition of potassium ferrocyanide to a suitably prepared solution and containing the copper salt with that obtained in a similar way from a standard solution. For more accurate work the quinosol method is recommended. Quinosol (the double sulphate of potassium and 8-hydroxyquinoline) forms a precipitate with copper salts according to the equation: $C_9H_8N(OH)\cdot SO_4K + CuSO_4 = C_9H_7N\cdot OCu\cdot SO_4K + H_2SO_4$. The precipitate is soluble in acids, hence the liberated acid must be neutralised, and the precipitate is then collected in a weighed Gooch filter. For confirmatory purposes the precipitate may be dissolved in strong sulphuric and nitric acids to destroy the organic nucleus, and the solution diluted and electrolysed, the copper being deposited on a weighed cathode. D. G. Hewer.

Change in caseinogen constituent in lime–caseinogen–calcium phosphate complex, and influence of rennin on this complex. Porcher.—See A., Aug., 795.

Patents.

Gelatin food. H. Thiele, Assr. to C. H. Boehringer Sohn (U.S.P. 1,586,129, 25.5.26. Appl., 13.7.25).—A composition for the manufacture of gelatin foods comprises gelatin together with lactic acid, free from anhydride, absorbed in a calcium salt of lactic acid. H. Moore.

Manufacturing yeast from malted milk. C. B. Hill and M. H. Givens, Assrs. to Northwestern Yeast Co. (U.S.P. 1,589,853, 22.6.26. Appl., 14.1.25).—Yeast is allowed to act upon a nutrient medium prepared by treating a gelatinised cereal with malt enzymes, milk is added, and the product is reduced to a dry state by using a sufficiently high temperature to destroy the fermenting power of the yeast. D. G. Hewer.

Preservation of sap-containing feeding-stuffs by the electric current. T. Schweizer (Swiss P. 106,942, 10.1.24).—An electric current of 2—4 amp. per sq. m. of electrode surface is sufficient, and reduces the risk of decomposition or oxidation during the process. B. W. Clarke.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Strength of digitalis preparations. IV. Effect of alcohol. C. de L. van Wijngaarden (Arch. Exp. Path. Pharm., 1926, 114, 21—31).—Exhaustive extraction of powdered digitalis leaves is obtained in the preparation of the 0·5% aqueous infusion of the Dutch Pharmacopœia and of the 10% tincture prepared with cold alcohol and in fractional extraction with cold water and 50% alcohol., *i.e.*, from the residues no appreciable amount of active glucoside can be subsequently extracted. Treatment of powdered digitalis leaves with alcohol vapour or warm alcohol, preferably by the so-called "stabilising" process causes an appreciable increase in the effective action of the digitalis powder (as measured by the lethal dose) and the preparations obtained on subsequent extraction or infusion are appreciably stronger. The effect is independent of the temperature at which the leaves are dried. More active solutions are obtained by Soxhlet extraction with absolute alcohol than by the infusion or tincture methods (cf. B., 1926, 607). Digitalis leaves which have been dried at a low temperature, *e.g.*, at 15° or 30°, show an increase of activity on keeping for a long period. R. Brightman.

Hydroxylamine hydrochloride for the quick determination of acetone. M. Marasco (Ind. Eng. Chem., 1926, 18, 701—702).—Rapid and concordant results can be obtained by the hydroxylamine hydrochloride method for the determination of acetone, using the following procedure and the factor 1·057. A sample of the liquid under test, containing not more than 0·2 g. of acetone, is run from a pipette into 400 c.c. of 0·2% hydroxylamine hydrochloride solution, neutral to methyl-orange. The mixture is titrated with standard alkali till nearly neutral, when it is stirred and after 20 sec. again titrated. The procedure is repeated until no appreciable amount of acid is liberated on keeping for 1 min., the end-point being determined by comparing the colour with a blank by light reflected from a white background. The method may be adapted to the determination of acetone in vapours by aspirating the gases under test through cold (15°)

hydroxylamine hydrochloride solution, or shaking a measured volume of the sample with the same reagent. Camphor does not affect the titration and alcohol up to 2·5% is also without effect. Some aldehydes, such as formaldehyde, may also be determined with hydroxylamine hydrochloride by this method. R. BRIGHTMAN.

Determination of alcohols and phenols in essential oils by means of magnesium methyl iodide.—T. ZEREVITINOV (Z. anal. Chem., 1926, **68**, 321—327).—All the alcohols and phenols that usually occur in essential oils react with magnesium methyl iodide with the evolution of methane equivalent to the hydroxyl groups present. Organic acids react similarly but the ketones which may be present, with the exception of acetone, are inert. To determine the alcohols and phenols in an oil, therefore, about 0·2 g., previously dehydrated over anhydrous sodium sulphate, is dissolved in 15 c.c. of anhydrous toluene or xylene and 5 c.c. of an ethereal solution of magnesium methyl iodide are added; the volume of methane evolved is calculated to hydroxyl and the hydroxyl due to the acids present (determined by titration of a second sample with potassium hydroxide) is deducted. From the remainder the percentage of alcohol or phenol is calculated. A. R. POWELL.

Erratum.—B., 1926, July 9, p. 565, col. 1, line 2 from top, *after* "cooled" *insert* "in a bath at a temperature."

Reactions of alcohols over zinc oxide catalysts. ADKINS and LAZIER.—See A., Aug., 807.

Reduction of carbon monoxide under ordinary pressure. JAEGER and WINKELMANN.—See II.

PATENTS.

Manufacture of derivatives [saturated or unsaturated dialkylamides] of pyridine-3-carboxylic acid. SOC. OF CHEM. IND. IN BASLE (E.P. 244,747, 5.12.25. Conv., 20.12.24).—The compounds are produced by the action of saturated or unsaturated dialkylamines upon quinolinic anhydride, a dialkylamine salt of quinolinic acid dialkylamide being produced at first, which on further heating splits up into pyridine-3-carboxylic acid dialkylamide, carbon dioxide, and the dialkylamine. For example, a mixture of 1 pt. of quinolinic anhydride and 3 pts. of diethylamine is boiled for 2 hrs. under a reflux condenser and excess of diethylamine is removed by distillation; the residue is heated to 180° until no more carbon dioxide is evolved, and pyridine-3-carboxylic acid diethylamide is recovered from the product by distillation *in vacuo*. The *diallylamide*, *methylpropylamide*, and *ethylpropylamide* of nicotinic acid are water-soluble oils having b.p. 147°/3 mm., 174°/16 mm., and 173°/15 mm., respectively. L. A. COLES.

Manufacture of hydrogenated di[hydr]oxydiphenylmethane compounds. CHEM. FABR. AUF. ACTIEN (VORM. E. SCHERING), and H. JORDAN (E.P. 252,594, 9.9.25).—Hydrogenated *pp*-dihydroxydiphenylmethane derivatives, of general formula $(HO\cdot C_5H_{10})(HO\cdot C_6H_4)CRR'$ and $(HO\cdot C_6H_{10})_2CRR'$, in which R and R′ are either the same or different aryl or alkyl groups, are obtained by the action of hydrogen in the presence of a catalyst on condensation products of a ketone with a phenol. Thus *pp′*-dihydroxydiphenyldimethylmethane (ββ-4:4′-dihydroxydiphenylpropane), from acetone and phenol, on hydrogenation at 170—180° and 10—15 atm. with a nickel catalyst (5 pts.) yields β-*p*-hydroxyphenyl-β-4-hydroxy*cyclo*hexylpropane, b.p. 213°/0·8 mm. In the presence of colloidal palladium at 50° complete saturation yields a mixture of bis-β-4-hydroxy*cyclo*hexylpropanes, b.p. 102—106°/12 mm., having an odour of lily of the valley and probably consisting of a mixture of *cis*, *cis-trans*, and *trans-trans* isomerides. At 150—170° in the presence of nickel bis-[β-6-hydroxy-*m*-tolyl] propane similarly yields *β-6-hydroxy-*m*-tolyl-β-4-hydroxy-3-methyl*cyclo*-hexylpropane*, b.p. 218°/0·8 mm., when the reaction is stopped after six atoms have been absorbed, and on complete saturation a mixture of stereoisomeric *bis-[β-3-methyl-4-hydroxy*cyclo*hexyl]propanes*, b.p. 108—112°/12 mm., having an odour of hyacinths. The condensation product of phenol and methyl ethyl ketone similarly yields a mixture of stereoisomeric *bis-[β-4-hydroxy*cyclo*hexyl]butanes*, b.p. 120—125°/14 mm. R. BRIGHTMAN.

Production of glucosides of *Digitalis*. C. H. BOEHRINGER SOHN, Assees. of F. RAHN (U.S.P. 1,586,116, 25.5.26. Appl., 13.7.25).—An aqueous extract of *Digitalis* leaves is extracted with butyl or amyl alcohol or other alcohol difficultly soluble in water, most of the alcohol is removed by evaporation under reduced pressure, and digitalein and gitalin are precipitated from the residue by addition of ether. The precipitate is treated with chloroform, in which gitalin alone is soluble, and one from which it is precipitated with ether or light petroleum. The glucosides may also be separated by removing gitalin from the aqueous extract with chloroform, and then recovering digitalein by extraction of the aqueous liquor with a higher alcohol. T. S. WHEELER.

Producing tuberculin. K. YOSHIZAWA (U.S.P. 1,586,937, 1.6.26. Appl., 11.5.25. Conv., 17.2.25).—Tubercle bacilli are cultivated in glycerol bouillon for two months and the culture, after sterilisation at 70°, is concentrated to one-tenth of its bulk, 70—80% of alcohol is added, and the liquid filtered after 24 hrs. The alcohol is evaporated off and the residual liquid is made slightly alkaline and digested with pancreatin at 37° until all the "tuberculin reactive constituent" as tested by Pirquet's or Römer's reaction has been destroyed. The product is fractionally sterilised at 63° to destroy the pancreatin and is then tested for bacilli by cultivation aerobically and anaerobically on glucose–agar for one week. The product is of therapeutic value in the treatment of tuberculosis especially skin tuberculosis and may be administered in doses 10,000 times as great as the normal tuberculin dose without injurious effects. T. S. WHEELER.

Basic phenol alkyl ethers. H. Hahl, Assr. to Winthrop Chem. Co., Inc. (U.S.P. 1,587,073, 1.6.26. Appl., 22.8.24. Conv., 21.9.23).—Phenols containing an unsaturated radical in the *o*-position to the hydroxyl group are treated with an alkaline reagent and a basic alkyl monohalide or a basic alkylene dihalide to yield the corresponding basic alkyl ethers, which are colourless or yellowish liquids of therapeutic value as substitutes for *Secale cornutum*, and form stable crystalline salts with acids, which are soluble in water and may be injected subcutaneously in aqueous solution. For example, 1·3 pts. of sodium dissolved in 50 pts. of alcohol are heated with 10 pts. of 8-hydroxy-7-allylquinoline and 10 pts. of β-diethylaminoethyl chloride until neutral, when the liquid is poured into water, and the oil obtained is separated, dried, and distilled. 8-β-*Dimethylaminoethoxy*-7-*allylquinoline* forms a yellowish liquid, b.p. 215—217°/20 mm. 2-β-*Diethylaminoethoxy*-3-*allylanisole* has b.p. 161—165°/12 mm., 2-β-*diethylaminoethoxy*-1-*allylnaphthalene* has b.p. 162—168°/10 mm., 2-β-*diethylaminoethoxy*-3-*crotonylanisole* has b.p. 174—178°/12 mm., and 2-β-*dimethylaminoethoxy*-3-*allylanisole* has b.p. 152—153°/12 mm. T. S. Wheeler.

Anthelmintic remedy. W. Kropp, W. Schranz, and W. Schulemann, Assrs. to Winthrop Chem. Co., Inc. (U.S.P. 1,587,077, 1.6.26. Appl., 29.11.24. Conv., 9.2.23; cf. G.P. 406,532; B., 1925, 474).—*o*-Benzylphenol is a valuable non-irritant anthelmintic, and its effects are enhanced by adding to it a substance which renders it liquid in the animal body. *p*-Benzylphenol which is formed in the manufacture of the *o*-compound is a suitable addition: it may be added to form 50—25% of the mixture. T. S. Wheeler.

Nitration process [for petroleum distillates]. M. B. Hopkins, Assr. to Standard Development Co. (U.S.P. 1,588,027, 8.6.26. Appl., 18.3.22).—1400 pts. of an octane fraction of petroleum boiling at 125—130° are mixed with 1000 pts. of 94% aluminium nitrate and 600 pts. of 68% nitric acid. The mixture is heated to 130—140° in an oil bath. The aluminium nitrate and nitric acid form a viscous layer on which the hydrocarbon floats. Hydrocarbon, nitric acid, and water distil off, the hydrocarbon being condensed and returned to the mixture. The resulting 950 pts. of reaction mixture are steam-distilled, yielding 370 pts. of crude mononitro-derivative and 580 pts. of unchanged hydrocarbon. The crude reaction product can be used as a solvent for cellulose derivatives and resins without separating. E. S. Kreis.

Preparation of di-substituted thioureas [thiocarbamides] of symmetrical structure. Silesia Verein Chem. Fabr., Assees. of Flemming & Klein Wissensch. Chem. Laboratorium (E.P. 244,070, 13.11.25. Conv., 6.12.24).—See U.S.P. 1,577,797; B., 1926, 464.

Synthesis of organic compounds [from carbon monoxide]. J. Y. Johnson. From Badische Anilin & Soda Fabr. (E.P. 254,819, 9.3.25).—See U.S.P. 1,562,480; B., 1926, 512.

Manufacture of alkyl halides. J. P. Wibaut (U.S.P. 1,591,151, 6.7.26. Appl., 2.1.24. Conv., 12.1.23).—See E.P. 209,722; B., 1924, 731.

Poly-iodinated isatins. Chem. Fabr. auf Actien (vorm. E. Schering), Assees. of W. Schoeller and K. Schmidt (U.S.P. 1,592,386, 13.7.26. Appl., 24.11.25. Conv., 10.12.24).—See E.P. 244,443; B., 1926, 514.

Manufacture of medicinal oil preparations [emulsions]. H. E. Potts. From Douglas Pectin Corp. (E.P. 252,476, 5.3.25).

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic solarisation. H. Belliot (Compt. rend., 1926, 183, 203—205).—Comparison of two identical solarised plates one of which had been developed and fixed and the other left as it was shows that to the solarised portions in the former there correspond blackened portions in the latter, a phenomenon which is absent in photographic inversion by the red and infra-red (cf. B., 1926, 693). With increase in the time of exposure the density of the solarised areas decreases, whereas that of the blackened areas increases. The wave-length producing maximum density for normal exposure is slightly greater than that producing maximum solarisation. R. Cuthill.

Thiocarbamide fog and a suggested explanation of Waterhouse reversal. S. O. Rawling (Phot. J., 1926, 66, 343—351).—The complexes formed by the action of thiocarbamides on silver bromide are decomposed in presence of low concentrations of alkali giving silver sulphide. The decomposition is hindered by the presence of soluble bromides. Assuming that silver sulphide particles on the grains in an emulsion can act as development centres, Waterhouse reversal is explained as follows: bromide set free by development of the latent image in a lightly exposed part of a plate suppresses the fogging action of thiocarbamide (in the alkaline developer) to such an extent that the sum of the densities of image and fog is less than that due to fog alone in an unexposed part of the plate. Comparing the fogging properties of an emulsion containing silver bromide–thiocarbamide complex with those of a similar emulsion containing silver sulphide, the curves representing growth of fog with time of development show in the former case the characteristic form due to the consecutive reactions (*a*) formation of silver sulphide and (*b*) development of grains infected by the silver sulphide. W. Clark.

See also A., Aug., 774, **Spectral sensitivity of chromates with organic substances** (Eder). 796, **Grain size and quantum theory of photographic exposure** (Hylan). 808, **Photochemical decomposition of silver iodide** (Hartung).

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Photographic silver halide emulsions. A.-G. FÜR ANILIN-FABR. (E.P. 246,800, 22.10.25. Conv., 31.1.25).—With the object of permitting a high speed to be obtained without the fog which normally accompanies prolonged ripening of an emulsion, a thiazole compound, *e.g.*, 0·125% of Thiazole Yellow, is added to the gelatin before preparing the emulsion. W. CLARK.

Sensitising photographic silver-halide emulsions for X-rays. R. SCHWARZ (G.P. 428,898, 13.11.25).—A thallium compound of low solubility, *e.g.*, thallous bromide, is added to the silver halide. The sensitivity is more then doubled. W. CLARK.

Copying drawings, printed matter, etc. by photography. A.-G. FÜR ANILIN-FABR. (E.P. 249,467, 17.9.25. Conv., 21.3.25).—An unfixed negative, developed on a non-hardened, slow emulsion of medium thickness, is washed and immersed in a solution of water, 1000 pts., potassium ferricyanide, 100 pts., ferric chloride, 20 pts., and potassium bromide, 15 pts. In this way there is formed on the silver-containing parts a loose deposit of Prussian blue, which prevents penetration of the hardening agent (ferric chloride), while in the unexposed parts of the negative the gelatin is hardened. On washing, the deposit is rinsed away from the exposed parts but adheres to the hardened unexposed parts owing to the mordant action of the ferric chloride. The plate is subsequently fixed and again washed. Alternatively, if the unexposed areas are relatively extensive, the plate is treated in a 25% solution of potassium ferrocyanide to convert the ferric chloride in the hardened parts into Prussian blue. The unhardened, exposed parts can be washed out with warm water, and the covering power of the hardened parts increased by dyes. Other suitable salts, *e.g.*, those of uranium, can replace the iron salts. W. CLARK.

Production of photographic image in colours on fabric. M. MICHEL (G.P. 427,505, 3.5.25).—The image produced on the material is developed with a tannic acid solution and the tannic acid salt thus produced is dyed in acid solution with basic dyes. Blue-print tracings are first bleached with an alkaline liquid. R. BRIGHTMAN.

Bleaching agent for the "Bromoil" process. E. MAYER (G.P. 426,661, 29.7.25. Conv., 1.12.24).—Betaine hydrochloride is added to the solution of copper sulphate, an alkali dichromate, and a soluble chloride, such as sodium chloride, or potassium bromide used as a bleaching agent in the "Bromoil" process. R. BRIGHTMAN.

Photomechanical printing surface and process. D. DE NAGY (U.S.P. 1,591,369, 6.7.26. Appl., 2.4.25. Conv., 4.4.24).—See E.P. 236,621; B., 1925, 786.

XXII.—EXPLOSIVES; MATCHES.

Laws of combustion of colloidal powders. H. MURAOUR (Bull. Soc. chim., 1926, [iv], 39, 841—846).—The general results of experiments on the combustion of insoluble nitroglycerin powders are recorded. The laws of the combustion are independent of the explosion temperature. Theoretically, the pressure is a logarithmic function of the explosion time, and it is shown that between explosion pressures of 500 kg. and 200 kg. below the maximum pressure, the experimental curve may be replaced by the theoretical logarithmic curve, the progression ratio ($q, = e^n$, where $e = 2·718$) being taken as the mean ratio for the experimental curve between the two points considered. The area bounded by the curve is inversely proportional to the thickness of the powder layer and is also approximately inversely proportional to the heat of decomposition of different powders under the same conditions of density and thickness. For the same powder the ratio (maximum pressure) $/n$ is constant, K, and the quotient $K/$(thickness of layer) is a constant depending on the chemical composition of the powder. The energy furnished per cm.^{2} of powder is constant for the same thickness. S. K. TWEEDY.

Influence of temperature on the energy of the combustion of powders. H. MURAOUR (Bull. Soc. chim., 1926, [iv], 39, 846—852).—The decomposition temperatures of nitroglycerin powders previously heated at 110° were obtained from the pressure–time curves determined by combustion in a bomb at ordinary temperatures and at 110° (cf. preceding abstract). Curves are also given connecting the energy of combustion with the preliminary heating temperature for powders decomposing at 250° and 340°; the possible mechanism by which the preliminary heating affects the energy of the reaction is considered. Nitrocellulose powders are less sensitive to temperature changes than nitroglycerin powders. S. K. TWEEDY.

Laws of combustion of colloidal powders. I. H. MURAOUR (Bull. Soc. chim., 1926, [iv], 39, 981—988).—As a preliminary to investigating the validity of Frankland's "layer" theory of combustion of explosive powders, the laws governing the combustion of such powders in closed vessels are investigated (cf. preceding abstracts). S. K. TWEEDY.

Nitration of various cellulose. G. MORIN (Mém. Poudres, 1926, 22, 57—68).—The possibility of replacing the varieties of cotton normally used in the manufacture of nitrocellulose by other cellulosic material was investigated during the war at the Angoulême powder factory. Among the sources of cellulose tried were various varieties of wood pulp, bamboo cellulose, jute, carex, rags, long-fibre cotton, cotton dust, blotting paper, and cotton which had been treated with sodium sulphite instead of with soda and chlorine. Tables are appended in which the purification, analysis, methods of nitration and stabilisation, stability and yield of nitrocellulose are given for each material. Cotton linters washed and bleached in the usual way can be replaced by linters washed in sodium sulphite or by rags and at least partly by certain washed and bleached celluloses such as those from pine and bamboo. S. BINNING.

Examination of nitrocellulose in polarised light. TISSOT (Mém. Poudres, 1926, 22, 31—56).—Nitrocelluloses with various nitrogen contents were obtained by nitrating cellulose with four types of mixed acid and were examined microscopically in polarised light. The colours of the nitrocelluloses varied with their nitrogen content, being yellow for the least nitrated and light blue for the most highly nitrated samples. The intermediate colours were violet-red and deep blue. Unnitrated cotton showed an iridescence which was absent from nitrocellulose. Examination in polarised light is suggested as a method for testing the homogeneity of samples of nitrocellulose and for estimating approximately the nitrogen content. To obtain consistent results certain precautions are necessary, the chief of which is the use of artificial illumination, preferably the Auer light. S. BINNING.

Waltham Abbey test for cordite powders. P. T. VIGNAU and R. T. BABUGLIA (Rev. Fac. Cien. Quim., 1925, 3, 47—50).—Contradictory results obtained by the Waltham Abbey test arise from variations in the details of the technique followed. It is apparently unnecessary to use silvered vessels for the test. The vessels and the powder should be absolutely free from moisture. Electrical heating and the recording of temperatures by means of a thermograph are desirable. G. W. ROBINSON.

Analysis of explosives. Apparatus for the 135° test. P. T. VIGNAU and J. ANGLI (Rev. Fac. Cien. Quim., 1925, 3, 51—53).—An apparatus for determining the stability of explosive powders at 135° is described. It is specially designed to avoid the loss of vapours and to minimise the effects of explosions. C. W. ROBINSON.

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Explosive. W. O. SNELLING, Assr. to TROJAN POWDER Co. (U.S.P. 1,588,277, 8.6.26. Appl., 11.12.24).—A filling composition for gas shells comprises nitrostarch, a liquid or solid toxic substance, such as diphenylcyanoarsine, and if desired a binder *e.g.*, lubricating oil, and an oxidising agent, *e.g.*, sodium nitrate. If the toxic substance is liable to decomposition by the heat of explosion a flame reducer such as oxanilide is added. Chlorinated hydrocarbons, such as carbon tetrachloride, may also be used; when the explosion takes place these are decomposed with formation of toxic compounds, such as phosgene. T. S. WHEELER.

[Smokeless] explosive mixture. A. E. SCOTT (U.S.P. 1,589,237, 15.6.26. Appl., 26.12.22).—Picric acid (16 pts.), alum (12 pts.), ammonium carbonate ($\frac{1}{3}$ pt.), and potassium chlorate ($\frac{1}{8}$ pt.) are dissolved in boiling water and the solution is cooled. The crystalline product which separates forms a safe, stable, practically smokeless blasting explosive. By varying the proportions of the ingredients it can be adapted for use in small arms or heavy ordnance. T. S. WHEELER.

Recovering nitric acid from waste acid (U.S.P. 1,590,043).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Use of aluminium hydroxide sol [in water purification]. E. W. JOHNSTON and F. P. DOWNEY (Canad. Eng., 1926, 50, [10], 94C—94D).—The successful use of aluminium hydroxide sol in the treatment of water is described. The sol was prepared by the method described by Coxe (B., 1923, 945 A). One grain of the sol per gal. reduces the alkalinity of the water 1 p.p.m. The analysis of the water being treated is as follows (in p.p.m.): total solidd 24, alkalinity (methyl-orange) 18, iron 3·4, carbon dioxide 3·5, hydrogen sulphide 2·0, oxygen consumes 8·0, colour 210—350. The p_H value of the water is 6·8, and it has a very strong odour and taste of hydrogen sulphide. Alum in amounts up to 10 grains per gal. and soda ash in amounts up to 4 grains per gal. failed to give a colourless effluent. Addition of the aluminium hydroxide sol in quantity corresponding to 6 grains of alum per gal. reduced the colour to 8 and the oxygen consumed to 0·5 p.p.m., and entirely eliminated the odour and taste. It is estimated that a saving of $21 per million gallons treated is effected by substituting aluminium hydroxide sol for aluminium sulphate. R. E. THOMPSON.

Colloid chemistry in [water] filtration. A. V. DELAPORTE and F. R. MANUEL (Canad. Eng., 1926, 50, [10], 94A—94C).—When "filter alum" is added to water the aluminium ions effect the precipitation of the negatively charged disperse phase and the sulphate ions coagulate the colloidal aluminium hydroxide subsequently formed. Carbonate ions, if present in sufficient concentration, also have a coagulating effect on the colloidal aluminium hydroxide and are adsorbed by the floc in the same manner as sulphate ions. Bacteria, colour, and organic matter are adsorbed by the colloidal hydroxide and are removed with the flocculent precipitate formed. R. E. THOMPSON.

Precise turbidity readings by electrical methods. L. H. SCOTT (J. Amer. Water Works Assoc., 1926, 15, 697—698).—The turbidity of raw influent water to a plant is ascertained by use of apparatus comprising a water tube, an electric lamp, a photo-electric cell, and a very sensitive galvanometer. A small portion of the influent water is made to circulate through the water tube, which is sealed at both ends with a convex lens. Under the lower lens the electric light is placed and at the top is placed the photo-electric cell in a light-tight compartment so arranged that the light from the tube will focus on the plate of the cell. The cell is made so that the resistance will vary in direct proportion as the square of the light thrown on it. As the turbidity changes the corresponding variation in the resistance is recorded on the galvanometer, the scale of which is calibrated to be read in turbidities. W. T. LOCKETT.

Reaction of *o*-tolidine with surface waters. O. FORSBERG (J. Amer. Water Works Assoc., 1926, 15, 706—708).—A number of surface waters containing iron and manganese gave a colour reaction with

o-tolidine. Waters containing only iron compounds when these do not exceed 10 p.p.m. do not react with o-tolidine, but waters containing even very small amounts of manganic hydroxide (0·5—2·0 p.p.m.), give a colour reaction similar to that produced with free chlorine. W. T. Lockett.

Cause and correction of red water troubles. T. Saville (Water Works, 1926, **65**, 75—84).—Increase in hydrogen-ion concentration due to carbonic acid does not influence the rate of corrosion until the p_H is less than 5·5, below which nearly all the free carbon dioxide is aggressive. Coagulation with alum, by increasing the free carbon dioxide, increases the corrosiveness of a water. A further effect of coagulation and filtration is the removal of organic matter, which may otherwise form a protective coating. This may explain the increase in corrosiveness of some waters following simple slow sand filtration. Waters coagulated with iron sulphate and lime are ordinarily non-corrosive. The corrosiveness of water is greatly increased by heating, due chiefly, it is believed, to decrease in viscosity, permitting an increased rate of diffusion of oxygen to the walls of the vessel, and also to convection currents which loosen the rust deposits. The addition of sufficient lime, soda ash, or caustic soda to produce a p_H of 7·0—7·5 will usually reduce corrosion to a minimum, although for complete removal of carbon dioxide a p_H of 8·5 is required. Protective coatings of lead will protect pipes from corrosion and will not cause lead poisoning when the p_H of the water is above 9—10. R. E. Thompson.

Manganese in the Amsterdam dune water supply. C. A. H. von Wolzogen-Kühr (Water en Gas, 1926, 31—34, 39—43; Water and Water Eng., 1926, **28**, 216).—Data are given on the occurrence of manganese in the mud and water of the Oranjekom and canals. The water contains 0·3 p.p.m. and the mud 0·1% of manganese, which is present as manganese dioxide in the latter case. The oxidation of manganese compounds to manganese dioxide, which undoubtedly occurs in the canals and rough filters, was shown to be due to manganese bacteria. Experiments with an artificially prepared solution of manganese carbonate in dune water showed that the oxidation of manganese compounds by the oxygen of the air does not take place at the p_H obtaining, namely, 8·1. Manganese bacteria were found to be capable of oxidising dissolved manganese not only in alkaline solution (p_H 10) but also in acid solution (p_H 4—5). Fresh gravel in rough filters at Leiduin became dark brown in course of time due to deposited iron and manganese oxides. The manganese oxide itself is also capable of absorbing manganese compounds from water. Fresh filters at first retain all manganese, then manganese begins to appear in the effluent, disappearing again in a few weeks. This is explained by the fact that development of the bacteria is insufficient to deal with all the manganese present until the latter stage is reached. The colour of manganese dioxide is less noticeable in fine sand filters owing to the relatively greater surface. R. E. Thompson.

Graphical chemistry in water softening. R. L. McNamee (Water Works, 1926, **65**, 285—290).—Nomographic and alinement charts are given for simplifying the solution of problems encountered in water analysis and water-softening investigations. R. E. Thompson.

Anti-microbial properties of various river and sea waters. Bacteriophagic powers. F. Arloing, Sempe, and Chavanne (Bull. Acad. Med., 1925, 43, [7]; Pub. Health Eng. Abs., 1926, W—8).—The anti-microbial power was found to vary in intensity and at times in specificity. It is usually greater in river water below cities, is increased or disturbed by floods, and is diminished and tends to disappear when the water is filtered through an L 3 filter candle and stored in the dark in a sealed tube for one month. Spring waters possess feebler bactericidal powers than polluted river waters. R. E. Thompson.

Rate of deoxygenation of polluted waters. E. J. Theriault (Publ. Health Rep., 1926, **41**, 207—217).—The "excess oxygen" method of determining the biochemical oxygen demand and the applicability of the results obtained to the study of stream pollution were investigated. Agreement between duplicate samples was excellent. After incubation for 16 days the rate of deoxygenation showed a marked acceleration and, contrary to the general view, appreciable quantities of oxygen continued to be absorbed even after 20 days. This cannot be attributed to approaching exhaustion of dissolved oxygen as the phenomenon has also been observed with fully aerated samples. Within wide limits, the rate of deoxygenation is independent of the amount of dissolved oxygen present. The evidence accumulated supports the hypothesis that under aerobic conditions the stabilisation of organic matter proceeds in two distinct and strictly consecutive stages; only after the carbonaceous matter is oxidised does nitrification set in. When only the first or carbon-oxidation stage is considered, Phelps' formula for the rate of deoxygenation gives values in reasonable agreement with experimental results. The 5-day values had a definite significance for relatively short periods of incubation, but after about 15 days' incubation the rate of deoxygenation increased markedly, the oxygen demand of the more highly oxidised sample being apparently twice as great as that of the same sample in a less highly purified state. The significance of this observation in the determination of percentage removal of organic matter by treatment plants is readily apparent, as in the extreme case in question the percentage values obtained would be in inverse relation to the purification actually accomplished. On the basis of nitrite, nitrate, and free ammonia determinations, it is probably safe to conclude that at 20° the oxidation of the purely nitrogenous impurities is virtually complete in 40—50 days. Appreciable quantities of dissolved oxygen, however, continue to be absorbed even after several months' incubation, probably due to slow oxidation of cellulose-like materials. After

30 days' incubation at 20° the rate of deoxygenation is practically constant. R. E. THOMPSON.

Rate of atmospheric re-aeration of sewage-polluted streams. H. W. STREETER (Pub. Health Rep., 1926, **41**, 247—262; cf. B., 1925, 565).—The re-aeration of flowing streams proceeds substantially in accordance with the physical laws governing absorption of gases. The rate of re-aeration is controlled mainly by the temperature, turbulence of flow, and the oxygen saturation deficit of the stream. An empirical method of measuring the rate of re-aeration is developed which gives results that appear to be consistent with known facts concerning the physical conditions influencing such rates, and by means of which reasonably accurate calculation may be made of progressive changes in the dissolved oxygen content of a stream under any given or assumed condition of flow, temperature, and initial pollution. The presence of sludge deposits and the density of pollution of the stream proper are important factors in determining the ability of a watercourse to recover its reserve supply of oxygen.
R. E. THOMPSON.

Investigation of pollution of Flambeau River at Park Falls. WISCONSIN RAILROAD COMMISSION (Pub. Health Eng. Abs., 1926, S.S.I.—11).—The evidence presented in a case concerning pollution of a stream by wood pulp and paper mill wastes is summarised. Reduction of dissolved oxygen below 2 p.p.m. for any material length of time will result in death of fish. Gas plant wastes and mine drainage are toxic to fish. R. E. THOMPSON.

Influence of soft and of hard waters on the decomposition of sewage. J. E. PURVIS (Surveyor, 1926, **69**, 277—278).—Sewage mixed with soft (distilled) water was oxidised at a much higher rate than when mixed with hard water (18·5—25°), the free and albuminoid ammonia and oxygen consumed values after aeration being lower in the former mixtures than in the latter. The nitrite content was irregular but the nitrates were invariably higher with the soft water. The hard water mixtures were also more opalescent in appearance, probably due to precipitation of colloidal matter. This would increase the amount of sludge deposited during tank treatment, thus reducing the load on the subsequent treatment processes employed. The importance of these factors in comparing the efficiency of plants and purification processes is emphasised.
R. E. THOMPSON.

Relation between ripe [sewage] sludge and incoming fresh solids. W. RUDOLFS, H. HEUKELEKIAN, and P. J. A. ZELLER (Amer. J. Pub. Health, 1926, **16**, 365—368).—A study of the influence of the rate of addition of fresh solids to digesting sludge in Imhoff tanks showed that when 2 pts. of fresh solids are added daily to 98 pts. of ripe sludge on the dry basis, digestion takes a normal course, while when 3 pts. are added digestion is impaired. With addition of 4% of fresh solids the sludge changes colour gradually from black to a peculiar greenish hue and later to yellowish-brown, becoming acid with an obnoxious odour. The experiments indicated that approximately 30 days are required for minimum satisfactory digestion under favourable conditions. Foaming appears to be primarily caused by an incorrect relation between old sludge and fresh solids.
R. E. THOMPSON.

Sewage disposal at Milwaukee. J. A. WILSON (Water Works, 1926, **65**, 115—121).—The plant consists of coarse bar screens, grit chambers, aeration tanks, and sedimentation tanks. The aeration period is 6 hrs. and the amount of air applied is equivalent to 1·5 cub. ft. per U.S. gal. of raw sewage. The effluent from the aeration tanks is retained in the sedimentation tanks for 30 min. and 90% of the settled sludge is returned to the incoming sewage. The sewage contains approximately 250 p.p.m. of suspended matter and the returned sludge increases this 10-fold. The remainder of the sludge is dewatered by filter-pressing. When the temperature of the sewage is above 20° the sludge is coarse enough to filter-press satisfactorily, but in winter when the temperature is as low as 7° chemical treatment is necessary prior to pressing. The relative filtering efficiency of the sludge is determined by filtration through Büchner funnels with the aid of vacuum. Treatment of February sludge with sulphuric acid to reduce p_H from about 7·8 to 3·4, the isoelectric point of plain sludge, increases the filtering efficiency, and addition of 0·1 g. of aluminium sulphate per 1 g. of dry solids in the sludge and adjusting p_H to 4·4, which has been found to be the isoelectric point of the mixture of alum and sludge, effects an additional increase in efficiency of about 50%, but the rate is still too low to give satisfactory results. The application of heat remarkably improves the filtering condition of both the acid- and the alum-treated sludge. The optimum temperature for acid-treated sludge is 88°, but the filtering efficiency of alum-treated sludge increases with rising temperature up to 100°. The sludge press-cake, after mixing with an equal quantity of previously dried sludge to prevent balling, is passed through Atlas direct-indirect-heat continuous rotary dryers, which reduce the moisture content from about 80% to 5—10% in 45 min. When mixed with potash and phosphate the sludge is a satisfactory complete fertiliser. R. E. THOMPSON.

Recent research in sewage disposal. F. A. DALLYN and A. V. DELAPORTE (Canad. Eng., 1926, **50**, 193—195, 213—215).—The work of the Experimental Station of the Dept. of Health of Ontario is reviewed. Digestion of activated sludge has been successfully carried out in a tank serving the combined function of a digestion chamber and storm flow tank. The iron content of activated sludge is of importance; a sludge of high iron content (5% as compared with 2%, dry basis) gives increased nitrification and clearer effluents, settles more rapidly, remains stable for fairly long periods, and has a higher nitrogen content. During an enforced period of inactivity of the aerating equipment of a small activated sludge plant, chlorine was successfully

used to reduce the odours due to putrefaction without interfering with the operation of the plant. The returned sludge has since been continuously heavily treated with chlorine without disturbing the purification process. Chlorine is a useful conditioning agent and sludge so treated does not putresce.
R. E. THOMPSON.

Combined determination of oxygen absorbed and albuminoid ammonia in sewages and effluents. J. W. H. JOHNSON (Analyst, 1926, 51, 345).—Ammonia is distilled off as usual, and to approximately 250 c.c. of liquid 25 c.c. of 20% sodium hydroxide solution and usually 20 c.c. of 0·125*N*-potassium permanganate solution are added. After heating under a reflux condenser for 30 min., and cooling, the solution is made up to 500 c.c. and two portions of 50 c.c. each are acidified, potassium iodide is added, and each titrated with 0·0125*N*-sodium thiosulphate solution. Each result gives the oxygen absorbed by one-tenth of the original volume of sample taken. The remaining 400 c.c. are returned to the flask, the ammonia is distilled off, and the distillate nesslerised; the albuminoid ammonia from four-fifths of the volume of sample taken is thus obtained. This is slightly greater than the ordinary Wanklyn value. D. G. HEWER.

Determinations of air dustiness with the sugar tube, Palmer apparatus, and impinger, compared with determinations with the konimeter. S. H. KATZ., G. W. SMITH, and W. M. MYERS (J. Ind. Hyg., 1926, 8, 300—306; cf. Greenberg, B., 1925, 1009).—The various forms of apparatus are described, and reasons given for the divergent results obtained with the same dust-carrying air. The ratio of the number of particles as determined by means of the konimeter to the numbers determined by the other methods diminishes as the amount of dust present increases. The relation is expressed by $y=ax^b$, where y=particles determined by the konimeter, x=particles determined by the other method, and a and b are constants for each kind of dust. Values of a and b are given for dusts of coal, grain, iron, limestone, silica, and granite.
S. I. LEVY.

Effect of turbulent air motion and of humidity on the stability of dust, fume, and smoke clouds. P. DRINKER, R. M. THOMSON, and (Miss) J. L. FINN (J. Ind. Hyg., 1926, 8, 307—313).—Turbulent motion has no effect on silica dust, a marked effect on fresh zinc oxide dust, and a considerable effect on tobacco smoke. Steam jets had a marked effect on silica and zinc oxide dusts. Increasing the humidity of the air before introducing the suspension caused zinc oxide to settle out more rapidly, but the effects on silica dust and tobacco smoke were inconsiderable.
S. I. LEVY.

Coal gas poisoning. DOLLINGER.—See II.

Prevention of lead poisoning in industry. KLEIN.—See XIV.

PATENTS.

Indicating the progress of chemical reactions [water softening]. R. S. MCNEIL, Assr. to COCHRANE CORP. (U.S.P. 1,587,782, 8.6.26. Appl., 3.1.24).—The softening of water by means of a zeolite-like material is controlled by leading off a small quantity of the treated water and treating it in a test-chamber with a liquid containing in solution a reagent which will form an insoluble precipitate with the substances causing hardness in the water. This precipitate settles and restricts the outflow of the water from the test-chamber, the height of the liquid in which is thus a measure of the efficiency of the softening process. When the zeolite is exhausted, the liquid level in the test-chamber rises to such an extent that a float closes the circuit of an electric indicating apparatus. E. S. KREIS.

Fumigant. G. H. BUCHANAN, Assr. to AMER. CYANAMID CO. (U.S.P. 1,586,175, 25.5.26. Appl., 19.1.25).—Hydrocyanic acid may not be capable of detection by its smell when it is present in the air in fatal quantities. To overcome this disadvantage it is mixed with 10—15% of cyanogen chloride, which is lachrymatory, and 2—4% of water for use as a fumigant. E. S. KREIS.

Preparation of sterilising agents, germicides, and the like. C. H. H. HAROLD, Assee. of UNITED WATER SOFTENERS, LTD. (U.S.P. 1,590,372, 29.6.26. Appl., 2.1.26. Conv., 23.3.25).—Chlorine solution is added to an ammonium salt solution, the ratio of ammonium radical to chlorine being 0·25—2 : 1 by weight. B. FULLMAN.

Purification of air [from ammonia] to render it suitable for breathing. G. ST. J. PERROTT and M. YABLICK (U.S.P. 1,586,327, 25.5.26. Appl., 31.10.25).—Finely-divided filling material is stirred into a hot concentrated solution of an organic or inorganic crystalline acid, so that the mass on cooling sets to a porous lumpy mass which will remove ammonia from the air. Boric acid is preferred. If the mass will not set of its own accord, a small quantity of hydraulic or oxychloride cement may be added. E. S. KREIS.

Softening water. F. SCHMIDT (U.S.P. 1,590,913, 29.6.26. Appl., 21.10.25. Conv., 12.1.24).—See E.P. 227,429; B., 1926, 302.

Treatment of household and street waste. K. GERSON (U.S.P. 1,593,491, 20.7.26. Appl., 26.11.24. Conv., 6.5.24).—See E.P. 247,284; B., 1926, 390.

Means for withdrawing and delivering measured quantities of liquids, applicable for delivering quantities of reagent solutions in water-treating apparatus. R. H. FROUDE, W. V. BOBY, and W. BOBY & CO., LTD. (E.P. 254,499, 22.6.25).

Filtering apparatus [for water]. B. BRAMWELL (E.P. 254,764, 11.2. and 9.11.25).

Insecticide (U.S.P. 1,589,866).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPTEMBER 17, 1926.

I.—GENERAL; PLANT; MACHINERY.

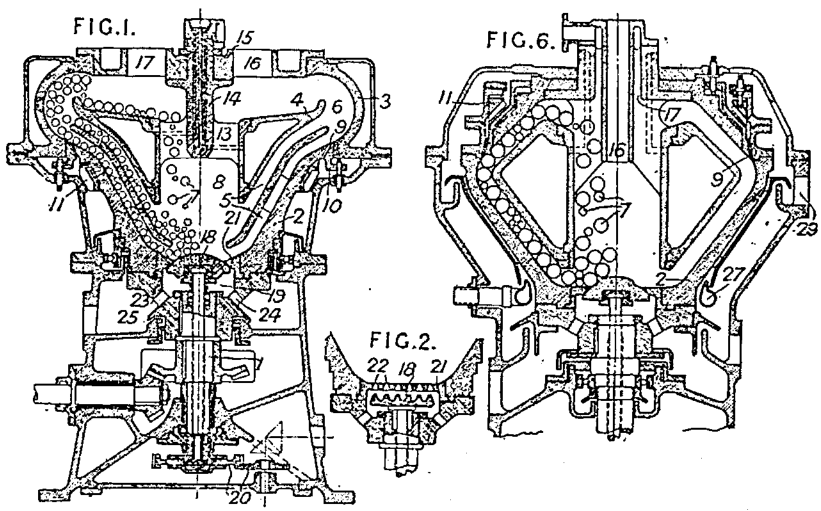
Aerosols in industry. W. E. Gibbs (J.S.C.I., 1926, **45**, 177—188 T).—Aerosols may be classified as dusts which fall with increasing velocity, clouds which ultimately fall with constant velocity, and smokes in which the particles show Brownian motion. Smokes and clouds are not formed by grinding, but only by condensation. Nearly all dusts become electrically charged as a consequence of collisions, the charges on large and small particles being opposite, or they may be charged by friction against a solid surface as in grinding. A cloud or smoke flocculates when in contact with a solid surface, and this process is accelerated by agitation or by increasing the area of the surface as in a filter. Smokes are less readily separated by filtration than gases, owing to the lower diffusion velocity of the particles. Thus smokes will pass a filter of wood charcoal, but are stopped by a paper filter, whereas a charcoal filter will remove chlorine quantitatively from air. The flocculation of an aerosol by electrical means proceeds by diminishing the attraction of a particle for its adsorbed gas. The efficiency of the removal of dust by settling, which is in itself scarcely practical for particles of diameter below 10^{-3} cm., can be improved by the application of centrifugal force as in the Calder–Fox scrubber or Pelouze tar-extractor, or by the weighting of the particles with water. Precautions against the risk of dust explosions are described. It is impossible to devise a respirator satisfactory in practice, and the only course is to prevent the liberation of a harmful dust so far as possible. C. Irwin.

Periodical heat-treatment of gas cylinders (Second report of the Gas Cylinders Research Committee, Dept. Sci. Ind. Res., 1926, pp. 29).—The effects of re-annealing and re-normalising overstrained specimens of 0·25% C and 0·45% C steel were determined. The specimens were annealed for 2 hrs. at 650°, the treatment therefore being similar to that used in practice for re-annealing gas cylinders. When slightly strained, to the extent of 1·5%, the structure of these steels tended to become one of ferrite and globular carbide, accompanied by a fall in the ultimate strength and by increased brittleness. Re-normalising produced no deleterious effects. Annealing more severely overstrained (6%) steel resulted in further deterioration in mechanical properties, but a single normalising treatment appeared to have a beneficial effect, though the original tensile strength was not reached. Surface hammering, representing ill-treatment of steel gas cylinders, did not produce any harmful effect on the metal, and it was concluded that the periodical heat-treatment of steel gas cylinders as recommended by the Home Office Committee of 1895 is not necessary. C. A. King.

See also A., Aug., 815, **Fine-pored filters and a new ultra-filter** (Zsigmondy); **Apparatus for automatically washing precipitates** (Hadjiev).

Patents.

Comminuting and mixing substances of all kinds. H. Hildebrandt (E.P. 234,520, 25.5.25. Conv., 26.5.24).*—Fig. 1 represents one form of a machine for grinding to a colloidal degree of fineness by means of balls which are caused to move



in a substantially different direction to the rotating part of the machine. The deep shell, 2, carried by the shaft, 1, is rapidly rotated and the centrifugal force causes the mixture of balls and material to climb spirally up the passages, 5, to the stationary grinding track, 3, where they lose their velocity and centrifugal force and return over the stationary guide, 4, down the central passage, 8. At intervals the valve, 18, is opened to allow ground material to fall out. The valve may be in the form of a grid as shown in Fig. 2. In the form shown in Fig. 6 the slot, 9, extends upwards, and heating or cooling liquid is supplied to the rotating shell wall by a nozzle ring, 27, and discharged through an opening, 29. The inlet opening, 16, and upper outflow opening, 17, are arranged centrally in this modification.

B. M. VENABLES.

Rod mill. R. C. GREENFIELD, Assr. to ALLIS-CHALMERS MANUF. Co. (U.S.P. 1,591,703, 6.7.26. Appl., 19.6.22).—A rotary casing entirely free from manhole openings is charged with rods nearly as long as the mill. The trunnion at the discharge end serves as a manhole, being made larger than 14 in., but not as large as one-third of the mill, in diameter.

B. M. VENABLES.

Ball mill. P. T. LINDHARD, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,591,916, 6.7.26. Appl., 15.8.25).—A ball mill is divided into primary and secondary grinding compartments between which is a separating compartment provided with a cylindrical screen. The material passes through perforations from the primary compartment to the interior of the screen, and the oversize not passing the screen is returned to the primary compartment by means, such as a conduit with internal helix, situated near the axis of the mill. Undersize which passes the screen is moved forward to the secondary compartment by means of conveyor flights on the cover of a handhole situated in the separating compartment.

B. M. VENABLES.

Comminuting mill. R. C. NEWHOUSE, Assr. to ALLIS-CHALMERS MANUF. Co. (U.S.P. 1,591,941, 6.7.26. Appl., 9.6.21).—A rotary drum is divided by transverse partitions into alternate grinding and non-grinding chambers. The partitions are perforated so that a gas blast can flow substantially straight through the whole mill; the perforations also permit the material to work through the mill. The non-grinding chambers are provided with lifters which cause the material to shower through the gas current.

B. M. VENABLES.

Determining the nature of the conditions which set up corrosion in plant traversed by flowing liquids. F. VON WURSTEMBERGER (E.P. 250,567, 30.3.26. Conv., 8.4.25).—Test-pieces of the same or different material as the plant itself, are attached to the plant and taken out at intervals for examination. The test-pieces should be used in pairs, one in metallic contact with, and the other insulated from, the body of the plant. B. M. VENABLES.

Apparatus for effecting intimate contact between liquids and gases. KIRKHAM, HULETT & CHANDLER, LTD., S. HERSEY, and F. W. STOKES (E.P. 254,382, 31.3.25).—The apparatus comprises a casing, divided horizontally into a number of compartments, in which the general flow of the gas is upwards while the general flow of the liquid is downwards. Each compartment contains a feed tray forming a reservoir of liquid and over this rotates rapidly a rotating tray provided with scoops depending from the bottom to pick up the liquid and with perforations in the circumference to allow it to pass out in the form of spray. The rotating trays in all compartments are supported on a common shaft, which is provided with mercury seals or glands at the ends. Further, each compartment is provided with an annular deflector extending inwards from the outer casing, thus causing the gases to move first inwards towards the tray, *i.e.*, against the spray, and then outwards, before rising into the next compartment. B. M. VENABLES.

Filter. F. F. FRICK (U.S.P. 1,590,233, 29.6.26. Appl., 5.5.23).—In a plate and frame filter-press, a diaphragm placed in each frame makes contact with the bottom and sides, but not with the top, interior peripheral surfaces. The diaphragm occupies the space not necessary for passage of pulp.

B. M. VENABLES.

Rotary drum filter. A. WRIGHT and F. W. YOUNG (U.S.P. 1,590,401, 29.6.26. Appl., 16.11.21). —A rotary filter is divided into a number of compartments each provided with an outlet pipe and a two-way cock arranged so that each compartment can communicate either with a common suction manifold or with the atmosphere. The drainage material behind the filter-cloths comprises wire woven into intertwined spirals. B. M. VENABLES.

Filter. E. L. OLIVER and J. F. BORDEN (U.S.P. 1,591,229, 6.7.26. Appl., 5.11.21).—The apparatus comprises a tank with filtering units suspended therein. The collected solid matter is periodically discharged back into the tank and the thickened pulp removed by a screw conveyer which is also provided with breakers for the filter-cakes.

B. M. VENABLES.

Dryer. W. M. BARKER (U.S.P. 1,590,798, 29.6.26. Appl., 31.7.24).—The material to be dried is flung by a rotating disc on to a depending annular wall, and drops through a funnel on to another disc, and so on down the apparatus. Spaces are left between the annular walls and the outer casing for the movement of a drying gas. B. M. VENABLES.

Centrifugal machine. C. A. OLCOTT, Assr. to S. S. HEPWORTH Co. (U.S.P. 1,591,004, 29.6.26. Appl., 3.6.25).—The stationary annular cover of a centrifugal machine is provided with means on its interior surface to cause any liquid tending to creep inwards towards the axis under the influence of the escaping air, to move outwards and rejoin the bulk of the separated liquid. B. M. VENABLES.

Catalytic reactions. P. Ellinger (G.P. 423,542, 8.12.22).—The catalytic power of base metals or their compounds is greatly increased by exposing the substances before or during the reaction in which they are used to the action of X-rays or the γ-rays from radioactive substances. Thus treatment of the cuprous chloride catalyst used in the Deacon-Hurter chlorine process enables a lower temperature to be employed with an increase in the conversion of hydrogen chloride to chlorine. The bleaching of rosaniline dyes by hydrogen peroxide in presence of potassium or sodium chloride or of aniline dyes by hydrogen peroxide in presence of copper sulphate is accelerated by treatment of the metal salts used as catalysts with X-rays. The oxidation of acetaldehyde to acetic acid by air in presence of ferrosoferric oxide is also accelerated by irradiation of the catalyst. T. S. Wheeler.

Evaporating liquids by atomising. J. H. Akkerman (G.P. 428,264, 26.10.24. Conv., 22.9.24).—The liquid or suspension is driven centrifugally over a number of plates or bands rotating in the same direction with the same angular velocity, and is partially evaporated. It is then atomised in a drying chamber in an air stream, warmed if necessary. The drying chamber is provided with apertures for admission of the drying agent and removal of the product, and with a number of plates or bands arranged in echelon on a common shaft at an angle of about 45°. A plane or curved plate is arranged on the axis over and near the admission aperture. L. M. Clark.

Rectification of liquids. O. Zitzke (G.P. 428,678, 27.1.25).—Concentric condensation chambers alternating with cooling partitions, discharge their contents into a common, central container. The condensate is removed and re-evaporated. The liquid to be rectified is led from a heated chamber over evaporating surfaces which are roughened, channelled, or covered with porous material. L. M. Clark.

Heat exchanger. G. D. Mantle, Assr. to Calorizing Co. (U.S.P. 1,593,537, 20.7.26. Appl., 28.5.24).—See E.P. 234,766; B., 1925, 976.

Apparatus for effecting transfer of heat between fluids. Aktiebolaget Ljungströms Angturbin (E.P. 243,723, 23.11.25. Conv., 28.11.24).

Heating gaseous fluids. H. A. Brassert and C. W. Andrews (E.P. 255,177, 29.4.25).

Heat interchangers. E. Lütschen (E.P. 255,364, 3.3.26).

Apparatus for exchanging heat in liquids or fluids. R. L. Munday (E.P. 255,711, 5.12.25).

Mixing concrete, mortar, chemical products, and the like. H. Ruef (E.P. 250,568, 30.3.26. Conv., 8.4.25).

Mixing of pulverulent materials. C. Pontoppidan (E.P. 255,375, 24.3.26).

Grinding and drying materials. Rheinische Maschinenfabr. (E.P. 252,200, 17.5.26. Conv., 15.5.25).

Refrigerating plant. E. Biaggini (E.P. 255,677, 17.9.25).

Filling bodies for heat-storage vessels, absorption towers and the like. Pfälzische Chamotte & Thonwerke (Schiffer & Kircher) A.-G., and O. Strack (E.P. 255,770, 11.3.26).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Definition of the terms coal, lignite, and peat. G. Stadnikov and N. Proskurnina (Brennstoff-Chem., 1926, 7, 197—199).—The adsorption of ferric hydroxide from a solution of ferric chloride by peat, lignite, and coal is suggested as a quantitative method of defining the fuels. 1 g. of peat, 1·5 g. of lignite, or 5 g. of coal are shaken for 5 min. with 75 c.c. of a 0·1% solution of ferric chloride and left for 1½ hrs.; the solution is then filtered and the concentration of ferric chloride determined by titration using potassium iodide and standard sodium thiosulphate solution. The amount of adsorption calculated as ferric oxide (Fe_2O_3) per kg. of fuel was below 4 g. for coals, 10—15 g. for lignites, and above 25 g. for peat in the particular fuels investigated. A. C. Monkhouse.

Degree of swelling of caking coals with particular reference to the geological and tectonic conditions of the coal seams. M. Dolch (Brennstoff-Chem., 1926, 7, 199—203).—The degree of swelling of a number of coals representative of different seams was determined using the crucible method (B., 1926, 347). Nickel and platinum crucibles gave the same results, but porcelain gave higher values. Particular coals showed variations in the degree of swelling, but average values for the same seam were in good agreement. The results obtained confirmed those of Fischer (cf. B., 1925, 233, 656) in correlating the solid and oily bitumen content with the geological age of the coal and its behaviour on coking. Coal ground to pass 225 meshes per sq. cm. gave more constant values than coarsely ground coal, the latter giving lower values. Washed coal on the average gave a lower degree of swelling than finely ground coal. A. C. Monkhouse.

Combustion of coal dust. R. von Walther and H. Steinbrecher (Braunkohlenarchiv, 1925, 9, 52—58; Chem. Zentr., 1926, II, 145).—Besides the ash content, grain size, and content of volatile matter, the chemical nature of the volatile matter and the position of the tar-forming zone play an important part in the combustion of powdered coal. In considering the explosiveness of combustible mixtures the quantity and condition of the evolved gas and the quantity of incombustible diluents should be considered. S. Pexton.

Coking of coals, and a proposed standard method for the determination of the agglutinating value of coal. M. Barash (J.S.C.I., 1926, **45**, 151—160 T).—The graded coal sample is intimately mixed with graded electrode carbon and the mixture (1 g.) carbonised under specified conditions at 800° in a small, vertical, silica tube placed in an electric furnace. The proportion of electrode carbon in the mixtures is varied until the carbonised product is just no longer coherent, and the agglutinating value of the coal is defined as the proportion of coal to electrode carbon (1 : x) in the limiting mixture. Starch (potato, rice, "soluble") may be used as agglutinant for standardising the electrode carbon. With the Lancashire coals examined the maximum agglutinating value lies at about 800°, and is diminished by atmospheric oxidation or extraction with solvents (*e.g.*, pyridine, chloroform). It is advisable to carry out a preliminary determination of the content of volatile matter in the coal sample at 800°, using the above-mentioned apparatus under specified conditions, and to examine the extent to which the product is swollen, as this will give an approximate indication of the agglutinating value of the coal.

W. T. K. Braunholtz.

Carbonisation in vertical retorts. (Fuel Res. Bd. Tech. Paper 15, 1926, pp. 45).—The investigation was made in order to determine whether Durham (Holmside) coal when carbonised in vertical retorts would yield gas with a calorific value of 560 B.Th.U. per cub. ft. and coke containing less than 3% of volatile matter. The best results were obtained when the baffle plate between the coal intake and the gas offtake was increased in length, the retort temperature was raised near the top, and no tar was circulated through the gas-collecting main. Under these conditions the gas yield per ton of coal was 66·7 therms with a calorific value of 568 B.Th.U. per cub. ft., and the coke yield per ton of coal was 14·87 cwt. containing 1·88% of volatile matter. A further series of tests was made to examine the effect of increased steaming on the carbonising results. These experiments were carried out with Holmside coal under the same conditions as those with other classes of coal (J.S.C.I., 1921, 333 R; B., 1923, 1163 A). Three tests were made, the steam admitted to the retorts being (*a*) 5·05, (*b*) 12·54, and (*c*) 20·21% respectively, by weight of the coal carbonised. The therms in gas, calorific value of the gas, and the coke yields were (*a*) 69·17, 524, 14·78, (*b*) 72·78, 504, 14·55, and (*c*) 76·51, 480, 14·49, respectively.

S. Pexton.

Ignition of firedamp by momentary flames. I. N. S. Walls and R. V. Wheeler. II. W. Rintoul and A. G. White (Safety in Mines Res. Bd., Paper No. 24, 1926, pp. 18).—I. An apparatus has been devised for bringing mixtures of methane and air into contact with a flame for measured small intervals of time, and has been used to determine the minimum time of exposure necessary to produce ignition in mixtures of varying composition. An explosion vessel was fitted with a metal cap over which a thin steel strip could be moved with a known velocity. The gas mixture in the vessel was exposed to a flame burning at a jet just above the steel strip during the time required for a hole in the steel strip to pass over a corresponding hole in the metal cap. Mixtures of methane and air containing between 9·5 and 10·0% of methane were most readily ignited, *i.e.*, required the shortest time of contact, which, for a small coal gas flame as igniting source, was about 7 milliseconds. The duration of exposure required for ignition is less, and the differentiation between one mixture and another with regard to ignitibility is less marked, the larger the volume of the flame used. If the flame used is not fully aerated, so that it can extract oxygen from the mixture to which it is exposed, the most readily ignited mixtures are those containing an excess of oxygen. Experiments on the ignition of mixtures of methane and air by flames from inflammable solids (strips of nitrated filter paper), and by flames from another explosive mixture, projected through an aperture of variable diameter, gave similar results, but were unsatisfactory as quantitative methods of investigation. From the fact that the mixtures of methane and air most readily ignited by fully oxidised explosives contain less methane than the mixtures most readily ignited by flames, it is suggested that the flame of an explosive is not solely responsible for its power to ignite gaseous mixtures. II. In a second form of apparatus for studying the ignition of firedamp mixtures a short cylindrical explosion vessel mounted at the end of a rotating spindle moved over a circular metal plate at a known velocity. At each revolution the lower open end of the vessel passed over a slot in the plate under which the flame of a mixture of hydrogen and methane with air was burning. For each flame used a certain number of mixtures of methane and air required about the same time of contact for ignition, but the range of such mixtures became narrower, and the mixtures contained less methane, as the oxygen deficiency of the flame was increased. The composition of the most readily ignitible mixture varied considerably with variation in the oxygen balance of the flame used for ignition. On the other hand, the temperature of the flame varied with the oxygen balance, and from this point of view a rise in the temperature of the flame decreased very considerably the time of contact necessary for ignition.

A. B. Manning.

Orifices of atmospheric gas burners. M. Kurokawa (J. Soc. Chem. Ind. Japan, 1926, **29**, 139—146).—The author has studied atmospheric gas burners with fixed orifices of the channel and sharp-edge types. The maximum jet efficiency is obtained with the channel type fixed orifice. For this type of orifice the formula $Q=1240\ KA(H/d)^{\frac{1}{2}}$ can be applied, where Q is the quantity of gas discharged in c.c. per sec. at N.T.P., K the discharge coefficient, A the sectional area of the orifice in sq. cm., H the orifice pressure in cm. of water column, and d the sp. gr. of the gas used. For a definite form of orifice, K is a constant independent of the value

of A and also of the quality of the gas. Therefore if the value of K for a particular form of orifice is previously determined and the sp. gr. and the supply pressure of the gas are known, the size of the orifice for any gas rate is easily calculated. The value of K generally lies between 0·6 and 0·95.

K. Kashima.

Determination of naphthalene in gas. L. S. Walters (J.S.C.I., 1926, **45**, 205—206 t).—The presence of indene in the picrates formed by passing coal gas from horizontal or vertical retorts through aqueous picric acid was established. For the determination of naphthalene in the presence of indene the gas is passed through the requisite amount of 33% acetic acid followed by a picric acid catch bottle. The contents of the acetic acid washers are mixed in a beaker and a slight excess of bromine dissolved in acetic acid is added to the solution, giving it a permanent yellow colour. The mixture is added to a large excess of saturated aqueous picric acid containing the picric acid used in the catch bottle. The indene, now in the form of the dibromide, is not precipitated by picric acid and the precipitate formed consists of pure naphthalene picrate.

S. Pexton.

Organic theories of oil origin. E. Clark (J. Inst. Petrol. Tech., 1926, **12**, 257—277).—Coal is considered to be derived from bog vegetation and oil from marine animals and plants. The kerogen content of oil shales is more closely related to coal than to oil. The conversion of the material of the organism into oil is attributed to the action of anaerobic bacteria; the effect of pressure-heat-time distillation is discounted to a large extent. Transference of oil from the mother rock to the reservoir rock is discussed.

W. N. Hoyte.

Liability to explosion of carburetted atmospheres in petroleum storage tanks. A. Williams-Gardner (J. Inst. Petrol. Tech., 1926, **12**, 336—340).—Samples were taken of the atmospheres existing in tanks used for the storage of petroleum and its products. Under no conditions was an explosive atmosphere found, the hydrocarbon percentage being above the explosive limit. This is shown to agree with theoretical requirements.

W. N. Hoyte.

Properties of paraffin wax. J. A. Carpenter (J. Inst. Petrol. Tech., 1926, **12**, 288—315).—From wax from Burmah crude oil a complete range of paraffins from $C_{21}H_{44}$ to $C_{34}H_{70}$ have been prepared. A paraffin with m.p. 96·5°, believed to be $C_{57}H_{116}$, was obtained in the Singu field, Burmah. Observations on the cooling of wax and its crystallisation from a variety of solvents point to the existence of a transition point between two isomeric crystalline forms for each paraffin at a temperature about 10—15° below the m.p. of the paraffin. Above the transition point acicular crystals are stable, below it rhombic plates are the stable form. Wax is more easily separated from mineral oil if acicular crystals are first formed by slow cooling, followed by rapid chilling. Dirt in suspension does not interfere with the crystallisation of wax, but asphaltic material and amorphous mineral jelly have harmful effects. Data for the thermal expansion and density of various waxes are tabulated. These confirm the existence of the transition points. Mottling of wax is due to the presence of mineral oil or dissolved air; wax may contain 7—10% by vol. of dissolved air. A breaking test is described which gives information on refining efficiency. Amorphous mineral jellies and crystalline waxes consist of two different chemical classes of compounds.

W. N. Hoyte.

Preparation of fuel oil by the distillation of the lime soap of soya bean oil. III. Experiments using oxides and carbonates of alkaline-earth metals as saponifying agent. M. Sato and K. F. Tseng (J. Soc. Chem. Ind. Japan, 1926, **29**, 109—115).—An intimate mixture of lime or calcium carbonate or magnesia or magnesium carbonate with the crude fatty acids of soya bean oil or with the oil itself may be used instead of the purified calcium soap of soya bean oil (B., 1922, 360 a; 1923, 758 a). The fuel oil is obtained rather more easily than by the earlier method, and so far as the properties, yield, and composition of the product are concerned, there is no notable difference between the two methods. The amount of saponifying agent used should be more than 20% of the oil or fatty acids. For equal amounts of saponifying agent the magnesium compounds produce a better fuel oil than the calcium compounds. With the carbonates of the metals a longer time is required for the distillation than with the oxides, but there is no noteworthy difference in the properties of the distillates. By using the retort residue of the distillation instead of fresh saponifying agents the distillation time is shortened, but the quality of the product is slightly inferior.

K. Kashima.

Coke-oven hydrogen for synthetic ammonia. Dodge.—See VII.

Utilisation of phenoxides from shale oil for wood preservation. Weiderpass and Kogerman.—See IX.

Patents.

Preparing raw peat to make it suitable for pressing. J. L. E. Sundling (E.P. 249,531, 17.3.26. Conv., 21.3.25).—Raw peat containing 90% of water can be rendered suitable for pressing by heating with an equal weight of a 2½—3% saline solution, such as sea water, to 60° for 10 min. The slurry loses about 90% of its water on pressing and yields a compact product containing 45% of water.

S. Pexton.

Agglomerating coal. F. Seidenschnur and H. Pape (G.P. 427,898, 6.3.25. Addn. to 427,271; B., 1926, 572).—Even the most fusible coals can be rapidly carbonised and agglomerated by distillation in a rapid stream of hot gases containing oxygen.

S. Pexton.

Composition and method of producing fuel. E. L. DAY (U.S.P. 1,592,758, 13.7.26. Appl., 5.1.25).—Charcoal is combined with tar during the process of distilling the latter in a liquid state in a closed vessel. H. ROYAL-DAWSON.

Combustion of ash-containing fuels. C. G. HAWLEY, Assr. to PEOPLE'S SAVINGS AND TRUST CO. OF PITTSBURGH (U.S.P. 1,591,679, 6.7.26. Appl., 15.1.21).—Air is caused to flow downwards with rotation within an annular chamber of refractory material situated above the place where the heat is to be utilised. Fuel is injected into the air stream and is burned partly in suspension and partly in contact with the refractory material; the flames and ash pass downwards into the lower chamber. B. M. VENABLES.

Operating horizontal chamber ovens. O. IMRAY. From N. V. SILICA EN OVENBOUW MIJ (E.P. 253,429, 30.11.25).—The level of the coal charge in an oven is varied according to the nature of the coal carbonised by the use of an adjustable levelling beam or two or more beams at different levels. A. C. MONKHOUSE.

Distilling bituminous fuels. G. PRITZBUER (E.P. 254,115, 24.7.25).—Fuel is brought rapidly to the distillation temperature by intense heating, the source of heat being at a much higher temperature than required for distillation. The fuel is moved from the hot zone to one of lower temperature where carbonisation continues because of the heat impetus received in the hot zone. This alternate high and low temperature treatment is repeated, and it is claimed that furnace fuel is economised and a very dense coke is obtained. The additional heat required for the high-temperature zones may be produced by electrical resistance heating, by high-frequency currents, or by induction currents. S. PEXTON.

Treating carbonaceous materials. W. E. TRENT (U.S.P. 1,589,022, 15.6.26. Appl., 27.9.24).—Coal is pulverised and mixed with oil, which forms a plastic mass with the carbonaceous matter and excludes the ash and water. This plastic mass is distilled so that the oil is recovered together with oil from the coal, and a portion of this oil is mixed with the carbonised coal to form a plastic or liquid fuel, whilst the remainder is used for treating a fresh quantity of coal. E. S. KREIS.

Cracking coal and oil. W. E. TRENT (U.S.P. 1,589,023, 15.6.26. Appl., 27.9.24).—The volatile portions of crude oil are vaporised in a heat exchanger, the heat liberated by the compression of the vapours being utilised. The residual oil is removed from the exchanger and used to mix with a suspension of coal in water, the water being heated to boiling with waste heat from the compressor. The agglomerates of coal and oil are moulded and baked, low-boiling oils being recovered from them during the baking process. E. S. KREIS.

Distillation of solid [bituminous] material. O. LASCHE, Assr. to KOHLENVEREDLUNG GES. (U.S.P. 1,590,895, 29.6.26. Appl., 15.11.23).—Bituminous fuel is moved slowly over an extended surface heated primarily by heat radiated from a massive heating body. S. PEXTON.

Recovering by-products from coal. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,591,729, 6.7.26. Appl., 18.3.22).—Heating gases are passed through certain passages formed in a briquette of pulverised coal, to vaporise the volatile matter, and the volatilised products are discharged from other passages not in communication with the passages receiving the heating gases. H. ROYAL-DAWSON.

Production of carbon monoxide. Production of water-gas. J. E. LEONARZ (U.S.P. 1,592,860–1, 20.7.26. Appl., [A] 10.9 and [B] 14.9.21).—(A) Carbon monoxide is prepared by heating iron oxide with carbon in the absence of air and passing the gas so formed through a bath of molten iron to which carbon is constantly added. (B) Water-gas is formed by passing steam through a bath of molten iron containing dissolved carbon. A. R. POWELL.

Manufacture of binding material and of moulded active carbons. E. GOUTAL and H. HENNEBUTTE (E.P. 241,899, 19.10.25. Conv., 24.10.24).—To obtain strong porous carbon, a binder is prepared by mixing coal tar and wood tar, together with oxides of iron, copper, or nickel or metal salts as catalysts, and heating the mixture to 200°; steam is evolved and a thickening of the tar occurs. The binder is mixed with a porous carbon, previously prepared, in proportions of 10—60%. The mixture is pulverised, compressed, and heated to 190—300° to remove water and then to 300—500°. A carbon of apparent density 0·7—0·9 is obtained which is readily combustible. If heated to higher temperatures the fuel is suitable for domestic heating or for forges. A. C. MONKHOUSE.

Activated charcoal. E. URBAIN (F.P. 603,806, 24.12.24).—Vegetable matter is extracted with a solution of zinc chloride, aluminium chloride, or ferric chloride, with or without addition of hydrochloric acid, or with sulphuric or phosphoric acid, and the insoluble matter is filtered off, dried, calcined, washed with hydrochloric acid, and again calcined at a temperature of at least 800°, when a very active adsorbent charcoal is obtained. The solution is evaporated and the residue carbonised with excess of zinc chloride, or alternatively it is washed free from zinc chloride and carbonised, with excess of calcium carbonate. The product when washed and pulverised forms a very good decolorising agent. T. S. WHEELER.

Carbonisation of wood. H. STRACHE and G. POLCICH (E.P. 253,206, 13.3.25).—Wood in pieces of 5—20 cm. diam. is carbonised above 600° in a vertical retort heated by producer-gas which after combustion and baffling to deposit soot is admitted at the middle of the retort and passes through the fuel bed. The charcoal falls into the

lower half of the retort where it is cooled by the circulation of distillation gases which may or may not contain tarry vapours. An alternative method is to use an internal producer, the retort being an annular shaft surrounding the producer. Carbon from the circulating gases is deposited on the charcoal; this effect may also be produced by impregnating semi-charcoal or wood with tar prior to carbonisation. A. C. MONKHOUSE.

Separation of oil from oil sands and like material. N. S. CLARKE, Assr. to ATHABASCA OIL PRODUCTS, LTD. (U.S.P. 1,592,179, 13.7.26. Appl., 5.7.23).—Oil sands are heated by means of a mixture of superheated steam and a suitable gas, and the heated product is mixed with water to separate the oil and solid material. C. A. KING.

[Motor] fuel. G. J. ESSELEN, JUN., Assr. to UNITED FRUIT CO. (U.S.P. 1,591,665, 6.7.26. Appl., 26.2.24).—The fuel consists mainly of ethyl alcohol and contains an alkali salt of a higher fatty acid and lubricating oil. S. BOWMAN.

[Motor] fuel. J. F. BEATTIE (U.S.P. 1,591,735, 6.7.26. Appl., 11.11.25).—The fuel consists of gasoline containing a small percentage of paraffin wax in solution. S. BOWMAN.

Production of motor fuel. SOC. ANON. "HYDROCARBURES ET DÉRIVÉS" (F.P. 602,434, 1.12.24).—Light tar oils are freed from water and phenols in known manner and then distilled. The vapours are condensed after passing over a mixture of coke and lime for the removal of sulphur compounds. The distillate is washed free from olefines and pyridine with concentrated sulphuric acid, neutralised with sodium carbonate, and finally rectified in contact with an anhydrous metal sulphate. S. PEXTON.

Manufacture of organic compounds [by catalytic reduction]. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABRIK (E.P. 255,127, 12.3.25).—Reducible organic compounds containing at least two carbon atoms in their molecules can be reduced at 250—600°, under a pressure of at least 50 atm., by gases containing hydrogen, with or without carbon monoxide, in presence of a contact mass containing one or more metal oxides which are not substantially reduced under the conditions of reaction. The mixtures of oxides are preferably made from oxides of metals of the first three periodic groups on the one hand, with oxides of metals of the fourth to seventh groups. Oxides of heavy metals capable of existing in several degrees of oxidation are suitable, but catalytic metals, or elements of the iron and platinum groups should be absent. By this process, unsaturated gaseous hydrocarbons are converted into oils resembling petrol, *m*-cresol yields toluene, and so on. A. DAVIDSON.

Treating [cracking] hydrocarbon oils. C. MATLOCK, Assr. to GASOLINE CORP. (U.S.P. 1,591,428, 6.7.26. Appl., 27.9.20).—A cracking still consists of a series of tubes, the outlets of which are connected to a common header. Means are provided for injecting a spray of cooling liquid into the header and thus diverting a mixture of vapours and condensed particles in a direction different from the general direction of the vapours and towards a dephlegmator column. S. BOWMAN.

Production of low-boiling hydrocarbons. STANDARD OIL CO., Assees. of F. M. ROGERS, M. G. PAULUS, and R. E. HUMPHREYS (Can. P. 254,448, 12.6.24).—Petroleum is heated to 390—430° in a closed vessel, and a vapour atmosphere containing at least 4% of the desired product of b.p. 120—200° is maintained. A light naphtha fraction boiling below 120° is formed. S. PEXTON.

Treating [dehydrating] hydrocarbons. R. CROSS (U.S.P. 1,591,744, 6.7.26. Appl., 22.12.21).—The method consists in intimately mixing the oil with bentonite and then separating the treated oil from the bentonite. S. BOWMAN.

Refining liquid hydrocarbons. HUILES, GOUDRONS ET DÉRIVÉS (F.P. 601,172, 22.10.24).—The crude product is freed from unsaturated compounds by distillation in the presence of 1—2% of anhydrous ferric chloride, zinc chloride, magnesium chloride, or aluminium chloride. S. PEXTON.

Lubricant. A. E. BECKER, Assr. to STANDARD DEVELOPMENT CO. (U.S.P. 1,590,800, 29.6.26. Appl., 9.3.22).—A lubricant which is a liquid at 150° and has a viscosity of not less than 49 sec. Saybolt is formed from 75—95 pts. of a fatty compound and 25—5 pts. of sulphur, combined at a temperature of 220°. B. M. VENABLES.

Low and medium temperature carbonisation of coal, oil shale, wood, peat, and the like. J. R. GARROW (U.S.P. 1,593,333, 20.7.26. Appl., 30.4.21. Conv., 31.7.18).—See E.P. 158,002; B., 1921, 172 A.

Carbonisation of coal. S. R. ILLINGWORTH, Assr. to ILLINGWORTH CARBONIZATION CO., LTD. (U.S.P. 1,593,697, 27.7.26. Appl., 22.5.26. Conv., 27.11.24).—See E.P. 248,077; B., 1926, 395.

Cooling of coke. S. R. ILLINGWORTH, Assr. to ILLINGWORTH CARBONIZATION CO., LTD. (U.S.P. 1,593,698, 27.7.26. Appl., 22.5.26. Conv., 27.11.24).—See E.P. 248,078; B., 1926, 395.

Fuels for internal-combustion engines. D. COSTAGUTA (E.P. 253,353, 4.6.25).—See F.P. 599,271; B., 1926, 430.

Converting heavy hydrocarbons into light hydrocarbons. F. B. DEHN. From UNIVERSAL OIL PRODUCTS CO. (E.P. 255,616, 29.6.25).—See U.S.P. 1,551,090; B., 1925, 951.

[Oil] still and method of distilling. P. MATHER (U.S.P. 1,594,209, 27.7.26. Appl., 9.8.21. Conv., 18.8.20).—See E.P. 175,666; B., 1922, 284 A.

Coal pulverising machines. A. HERBERT and C. E. BLYTH (E.P. 255,187, 16.5.25).

Gas-producer furnaces. L. RAINCHON (E.P. 255,329, 15.1.26).

Gas retort settings. G. R. HISLOP (E.P. 255,637, 16.7.25).

Bituminous emulsions (E.P. 254,004 and 254,012).—See IX.

Bringing about changes in dielectric carbon compounds (E.P. 254,375).—See XI.

Partial oxidation of gaseous hydrocarbons (U.S.P. 1,588,836).—See XX.

III.—TAR AND TAR PRODUCTS.

PATENTS.

Motor fuel (F.P. 602,434).—See II.

Manufacture of organic compounds by catalytic reduction (E.P. 255,127).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Adsorption of Methylene Blue by zinc oxide. H. M. BURNS and J. K. WOOD (J. Soc. Dyers and Col., 1926, **42**, 210—212).—Samples of zinc oxide prepared by treating a 10% solution of zinc acetate with a large excess of caustic soda, passing carbon dioxide through the solution of sodium zincate, and washing the precipitated zinc oxide until the washing water had p_H 7·4, and by treating a 10% solution of zinc acetate with caustic soda until the precipitate of zinc oxide just began to re-dissolve and then washing it until the washing liquors had p_H 6·3, had no adsorptive power for basic dyes but readily adsorbed acid dyes. Samples of zinc oxide prepared by the second method and washed until the washing waters had p_H 8·5, 8·3, 7·8, 7·4, 7·2, and 6·5, respectively, adsorbed quantities of acid dyes which increased in the order named, but the first four samples adsorbed quantities of basic dyes (Methylene Blue and Magenta) decreasing in the order named and the last two samples had no affinity for basic dyes; the acid dyes could not be removed, but the basic dyes were completely removed by washing with water. The presence of phosphates causes zinc oxide to adsorb Methylene Blue from aqueous solutions having p_H 6·3 when the concentration of the phosphate ion exceeds $M/100$—$M/150$, but the corresponding effect of hydroxyl ions is much greater; the dyeing properties of zinc oxide are therefore influenced in the same manner as those of the textile fibres (cf. Briggs and Bull; B., 1923, 91 A). Zinc oxide dyed with basic dyes in the presence of hydroxyl ions could be completely freed from dye by washing with water.

A. J. HALL.

Modification of the Höchst method for the determination of anthracene. F. JACOBSOHN (Chem.-Ztg., 1926, **50**, 545—546; cf. B., 1925, 838).—The time required for the Höchst test may be shortened to 8 hrs. by the use of a glass filtration apparatus in place of the filter-paper and Gooch crucible of the original method. Higher results are also obtained in consequence of reduced mechanical losses. The following procedure is recommended. The sample of anthracene is oxidised with 5—25 c.c. of chromic acid solution for ½—1 hr., then cooled and diluted. The anthraquinone is collected in a Jena glass filter-crucible filter-plate, 4 cm. diam.; (the 3 cm. diam. size can also be used at this stage, but not subsequently). After washing with ice-water, hot 1% sodium hydroxide, and hot-water, the crucible and washed precipitate are dried at 110° for ½ hr. and warmed in a flat-bottomed lipped glass evaporating dish; 10 c.c. of 10% oleum are added, and the mixture is heated for 10 min. at 100—110°. The anthraquinone dissolves completely and drops through the filter-plate into the dish. The crucible is washed twice with 2·5 c.c. of concentrated sulphuric acid and the solution is poured out of the dish into cold water (volume with washings =300 c.c.). After ¼ hr. the anthraquinone is collected in a glass filter-crucible with filter-plate 4 cm. or 6 cm. diam., washed as before, dried at 110°, and sublimed at 250—350°. With the filter-plate of 6 cm. diam. the operations can be completed in 6 hrs. The method is based on experimental work by Eller and K. Koch.

R. BRIGHTMAN.

See also A., Aug., 830, **α-Naphthylcarbimide as reagent for phenols and aromatic amines** (FRENCH and WIRTEL). 839, **Action of sulphite on 1:5- and 1 : 8-dinitro-2-methylanthraquinone** (EDER and MANOUKIAN). 840, **Violanthrone dyes from benzofluorenones** (GEORGEACOPOL). 845, **2:5-Diaminopyridine** (TSCHITSCHIBABIN and POZDNIAKOV). 848, **2-Phenyl-αβ-naphthatriazolequinone** (CHARRIER and MANFREDI). 850, **Thiazole dyes** (YAMADA).

Catalytic oxidation of organic compounds in the vapour phase. DOWNS.—See XX.

PATENTS.

Manufacture of new azo-dyestuffs. I. G. FARBENIND. A.-G. Assees. of FARBENFABR. VORM. F. BAYER & CO. (E.P. 245,765, 2.1.26. Conv., 8.1.25).—New azo-dyes giving yellow shades on wool, faster to fulling, perspiration, and light and superior to those produced by known pyrazolone-azo-dyes are obtained by combining an aromatic diazo-compound with a methylpyrazolone or a pyrazolone-carboxylic acid derived from an aminodiarylsulphone. When applied as lakes the new dyes are characterised by superior fastness to liming and to water and light. The new pyrazolones can also be used as components for chrome dyes for wool, giving with *o*-hydroxycarboxylic acid derivatives very fast greenish-yellow or red dyes, fast to potting. Thus *4′-methyl-2-aminodiphenylsulphone-4-sulphonic acid*, obtained by reducing the condensation product of 4-toluene-1-sulphinic acid and 1-chloro-2-nitrobenzene-4-sulphonic acid, when converted into the hydrazine and condensed with ethyl acetoacetate by the usual methods, yields a *methylpyrazolone*, affording with diazotised *o*- or *p*-sulphanilic acid in the presence of sodium acetate *dyes* giving greenish-yellow shades

on wool, whilst with diazotised 4-chloro-2-nitroaniline in the absence of sodium acetate, a *dye* is obtained giving reddish-yellow size colours, fast to lime and light. If the hydrazine of 4′-methyl-2-aminodiphenylsulphone-4′-sulphonic acid is condensed with ethyl oxaloacetate there is obtained a *pyrazolone-carboxylic acid*, 438 pts. of which combined with the diazo-compound from 327 pts. of 4′-methyl-2-aminodiphenylsulphone-4-sulphonic acid in the presence of sodium acetate affords a *dye* giving greenish-yellow size colours. A similar *dye* is obtained with diazotised *4′-methyl-2-aminodiphenylsulphone* or by combining with the *pyrazolone-carboxylic acid* obtained from the analogously prepared *4′-methyl-4-aminodiphenylsulphone-2-sulphonic acid.* Similarly *2-amino-4′-hydroxydiphenylsulphone-4-sulpho-3′-carboxylic acid* (from salicylic-sulphinic acid and 1-chloro-2-nitrobenzene-4-sulphonic acid) affords a *pyrazolone*, 454 pts. of which combined with 173 pts. of sulphanilic acid in the presence of sodium acetate, give a greenish-yellow *dye*, the shade of which is not appreciably altered by after-chroming. By combining with 143·5 pts. of 4-chloro-2-aminophenol in alkaline solution, the same pyrazolone affords an orange *dye*, giving fast red shades on after-chroming.

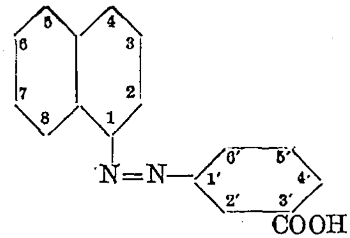
R. BRIGHTMAN.

Manufacture of substantive green azo dyestuffs. A.-G. FÜR ANILIN-FABR., Assees. of W. LANGE (G.P. 420,818, 22.2.24. Addn. to 419,585; B., 1926, 432).—The components specified in the chief patent are coupled in the following way:—The unsymmetrical carbamide obtained from a yellow aminoazo-compound and an aminohydroxy-compound of the naphthalene series by treatment in aqueous solution with phosgene, is coupled with the diazo-compound of a secondary disazo-dye obtained from a first component and two middle components not containing hydroyxl-groups.

Manufacture of a vat [anthraquinone] dyestuff. SOC. OF CHEM. IND. IN BASLE (E.P. 249,099, 26.2.26. Conv., 10.3.25. Addn. to 231,555; B., 1925, 437).—The dye described in the earlier patent is now obtained by treating a 1-halogen-2-amino-anthraquinone-3-carboxylic acid with an agent which eliminates hydrogen halide. Thus 173 pts. of 1-bromo-2-aminoanthraquinone-3-carboxylic acid (from 2-aminoanthraquinone-3-carboxylic acid and bromine in acetic acid), 15 pts. of anhydrous sodium acetate, 0·5 pt. of copper chloride, and 1700 pts. of nitrobenzene are boiled together, with stirring, under a reflux condenser. The mixture is cooled and filtered and the solid product washed successively with toluene, alcohol, and water. The crude dye is then boiled with dilute sodium carbonate to remove unchanged parent material and purified in known manner.

R. BRIGHTMAN.

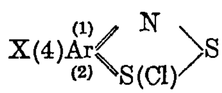
Production of benzanthrone derivatives. G. KALISCHER, R. MÜLLER, and F. FRISTER (U.S.P. 1,588,376, 8.6.26. Appl., 17.8.25. Conv., 24.12.24).—α-Naphthaleneazobenzene-*m*-carboxylic acids of the general formula



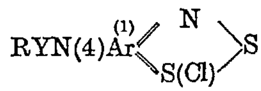
in which the C-atoms 4, 5, and 4′ are not substituted, are reduced in acid solution to the corresponding hydrazo-compounds which are simultaneously subjected to the benzidine transformation. The 4 : 4′-diaminophenylnaphthyl-2′-carboxylic acids obtained are tetrazotised, and the tetrazo-compounds are treated by known methods to replace the original amino-groups by hydrogen, halogen, hydroxy, methoxy, etc. The products are subsequently condensed with acid condensing agents to the corresponding benzanthrone derivatives. The products are of value as intermediates in the preparation of vat dyes of the Violanthrone and the *iso*Violanthrone series. The α-naphthaleneazobenzene-*m*-carboxylic acids are obtained by boiling with alcohol the diazo-compounds of the azo-dyes formed by coupling diazobenzene-*m*-carboxylic acid with α-naphthylamine or its derivatives. Suitable acid condensing agents for the last stage comprise chlorosulphonic acid, sulphuric acid, or zinc chloride; alternatively, the compound is converted into the corresponding acid chloride and treated with aluminium chloride. As an example the compound from *m*-diazobenzoic acid and α-naphthylamine is diazotised and boiled with alcohol to give α-naphthaleneazobenzene-*m*-carboxylic acid, which is reduced to the corresponding hydrazo-compound with stannous chloride in hydrochloric acid, when transformation simultaneously occurs. The 4 : 4′-diaminonaphthylphenyl-2′-carboxylic acid is tetrazotised and treated with cuprous chloride to yield the corresponding dichloro-compound which is dissolved in chlorosulphonic acid below 10°; when the solution is poured on to ice *4 : 4′-dichlorobenzanthrone*, m.p. 215°, separates.

T. S. WHEELER.

Quinone vat dyestuff, dyeing particularly animal fibres in the vat fast shades. R. HERZ, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,588,384, 8.6.26. Appl., 13.5.24. Conv., 19.5.23).—Compounds of the type described in E.P. 17,417 of 1914 (B., 1921, 619 A), which may be obtained by the action of disulphur dichloride on certain aromatic amines, and which are probably arylenethiazthionium chlorides of formula (I.)



(I.)



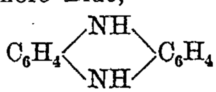
(II.)

in which X indicates a labile substituent such as chlorine or an alkoxy-group, are condensed with primary or secondary arylamines to give compounds of formula II. in which R denotes aryl which may or may not contain substituents and Y indicates

hydrogen or an aryl or aralkyl group. By the action of alkalis with or without addition of reducing agents on these products the heterocyclic ring is ruptured and the corresponding arylamino-derivative of an *o*-aminoarylmercaptan is obtained. These compounds condense with arylquinones or their halogen-substituted derivatives to give compounds containing probably a thiazine nucleus which dye fibres, especially animal fibres, in the hyposulphite vat shades which are fast to washing, milling, and light. In place of the mercaptan compounds their chemical equivalents which will form them during the reaction may be used, *e.g.*, the corresponding *o*-aminoarylthiosulphonic acids or *oo*-diaminodiaryl disulphides, whilst the quinones may be replaced by the corresponding quinols if an oxidising agent is added during the condensation. As an example 4-chloro-6-methyl-1 : 2-phenylenethiazthionium chloride (from *o*-toluidine and disulphur dichloride) or 4-methoxy-6-methyl-1 : 2-phenylenethiazthionium chloride (from disulphur dichloride and 2-amino-5-methoxytoluene) is condensed with aniline to give 4-phenylamino-6-methyl-1 : 2-phenylenethiazthionium chloride. The latter is treated with alkaline hyposulphite, and the zinc derivative of 6-*amino*-3-*phenylamino*-5-*methylphenyl*-1-*mercaptan* obtained by addition of zinc chloride, is condensed with chloranil by boiling in acetic acid for some hours, to yield a dye which gives fast bright violet-blue shades on animal fibres. Using quinone in place of chloranil a product giving more reddish shades is obtained. 4-Phenylamino-6-methoxy-1 : 2-phenylenethiazthionium chloride from disulphur dichloride and *o*-anisidine when condensed with aniline, gives on reduction and treatment with chlorobenzoquinone a dye which yields indigo blue to greenish-blue shades. Replacement of *o*-anisidine by α-naphthylamine and of chlorobenzoquinone by β-hydroxy-α-naphthaquinone or 2 : 3-dichloro-α-naphthaquinone in the above example yields a product which gives grey-blue to black shades.

T. S. WHEELER.

Indigo dyestuff. S. N. GUPTA (U.S.P. 1,588,960, 15.6.26. Appl., 10.5.24).—Aniline is oxidised in acid solution with, *e.g.*, chromium trioxide, barium peroxide, potassium chlorate, or calcium hypochlorite to give Chromophore Blue,



This is treated with an organic acid such as acetic, citric, oxalic, or tartaric acid, capable of yielding the grouping, CO·C : C·CO, whereby indigo is produced.

T. S. WHEELER.

Manufacture of 2 : 3-aminonaphthoic acid. SOC. OF CHEM. IND. IN BASLE (E.P. 250,598, 7.4.26. Conv., 8.4.25).—Improved yields of 2 : 3-aminonaphthoic acid are obtained by heating 2 : 3-hydroxynaphthoic acid in a closed vessel with aqueous ammonia in the presence of an ammonia double compound, such as zinc or calcium ammonium chloride. For instance, a solution of 250 pts. of zinc chloride in 300 pts. of water is mixed in a stirring autoclave with 500 pts. of 2 : 3-hydroxynaphthoic acid. The autoclave is closed, 120—140 pts. of gaseous ammonia are introduced, and the autoclave is heated at 220—230° (12—20 atm.) for 24 hrs. The thick yellow magma produced is boiled with 4000 pts. of water and 1400 pts. of concentrated hydrochloric acid, filtered hot, and the filtrate mixed with 1200—1500 pts. of common salt. The hydrochloride of 2:3-aminonaphthoic acid separates on cooling. The crystals are washed with saturated brine, dissolved in 7000 pts. of 8% sodium carbonate solution, filtered from insoluble matter, and the 2:3-aminonaphthoic acid precipitated by feebly acidifying the solution. The yield is about 80% of theory. R. BRIGHTMAN.

Producing amino[hydr]oxy-compounds of the aromatic series by electrolysis. H. E. POTTS. From CHEM. FABR. GRÜNAU LANDSHOFF & MEYER (E.P. 254,204, 29.12.25).—Increased yields of amino-hydroxy-compounds of the aromatic series may be obtained by the reduction of nitro-compounds by means of base metal cathodes by adding the nitro-compound gradually to the catholyte in the presence of a colloid or a substance acting as a colloid, using a current density greater than 6 amp./dcm.^{2}. For example, nitrobenzene is added to a catholyte of sulphuric acid and glue enclosed, together with the copper cathode, in a diaphragm which is immersed in a lead vessel (anode) containing sulphuric acid of 30° B. (*d* 1·26). Electrolysis is carried out with a voltage 3—3·5 volts, current density 15 amp./dm.^{2}, at 80—90° ; 7 kg. of nitrobenzene yield 4 kg. of *p*-aminophenol. B. FULLMAN.

Manufacture of naphthylamine sulpho-acids. E. F. EHRHARDT and R. M. HEREWARD (E.P. 254,402, 4.4.25).—1 : 4-Naphthylaminesulphonic acid together with 1 : 5-naphthylaminesulphonic acid is obtained by treating α-nitronaphthalene with 3 mols. of sodium sulphite or a mixture of sodium sulphite and ammonium sulphite in the presence of excess of ammonia at 130—140°. Ammonium sulphite alone does not give such good results as when mixed with sodium sulphite. The yield compares favourably with that from α-naphthylamine sulphate. For instance, 109 g. of α-nitronaphthalene are treated with 600 c.c. of strong sodium bisulphite solution neutralised with 395 c.c. of ammonia liquor (*d* 0·88). The nitronaphthalene is charged into the autoclave first, then the ammonia and the bisulphite last. The mixture is stirred at 130—140° for 4 hrs., when the liquid is filtered, the excess of ammonia recovered, and the solution acidified and boiled to drive off sulphur dioxide. The naphthionic acid is filtered off hot and from the mother liquors the 1 : 5-naphthylaminesulphonic acid separates on cooling. The crude naphthionic acid is purified by suspending in boiling water and again filtering.

R. BRIGHTMAN.

Production of azo dyestuffs. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 250,075, 13.7.25).—See U.S.P. 1,573,605 ; B., 1926, 432.

Azo dyestuffs containing zinc. SOC. OF CHEM. IND. IN BASLE, Assees. of F. STRAUB and H. SCHNEIDER (U.S.P. 1,572,573, 9.2.26. Appl., 1.10.25).—See E.P. 241,572; B., 1926, 576.

Vat colouring matter. BADISCHE ANILIN- & SODA-FABR., Assees. of A. LÜTTRINGHAUS and A. WOLFRAM (U.S.P. 1,582,844, 27.4.26. Appl., 8.6.25).—See F.P. 599,038; B, 1926, 577.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of the pure wool content of raw wool. E. SCHÜLKE (Textilber., 1926, 7, 121).—The nitrogen content of the residue obtained by removing from raw wool all adhering nitrogenous impurities is an accurate indication of the content of pure wool, which contains 13—16% N. For technical purposes a 30-g. sample taken from 1 kg. of mechanically disintegrated raw wool is thoroughly washed with hot water, filtered, the residue dissolved in 20 c.c. of warm concentrated sulphuric acid in a tared porcelain dish, and powdered gypsum added (with stirring) till the mixture weighs 100 g. Subsequently the nitrogen content of 10 g. of the mixture (equivalent to 3 g. of raw wool) is determined by the Kjeldahl method and the pure wool content calculated from the result. Alternatively the washed and filtered wool is further purified by washing with trichloroethylene or carbon tetrachloride, then moistened with 3% of sulphuric acid, dried (carbonised), washed with water, dried to constant weight at 110°, and the residual pure wool weighed.

A. J. HALL.

Swelling and mercerisation of natural cellulose fibres in nitric acid, and "philanised" cotton. I. *X*-Ray spectography. J. R. KATZ and K. HESS (Z. physikal. Chem., 1926, 122, 126—136; cf. B., 1925, 495).—If fibres of ramie, flax, or jute are subjected to the action of 69% nitric acid for at least 1½ hrs., they give the same *X*-ray interference diagram as fibres which have been mercerised with sodium hydroxide. With acid concentrations below about 61·3% the diagram is the same as that of ordinary cellulose, and no swelling of the fibre occurs. Knecht's compound of cellulose and nitric acid (B., 1904, 335) gives a diagram which differs from that given by cellulose, whereas "philanised" cotton (stated to be prepared by a short treatment with concentrated nitric acid) gives a diagram practically identical with that of cellulose.

R. CUTHILL.

Is cellulose acetate silk superior to German [cellulose] silks? E. SCHÜLKE (Textilber., 1926, 7, 25—26).—A comparison of various types of artificial silk shows that the dry and the ratio of wet/dry strengths (in g. and calc. to 150 denier) of viscose, Vistra, cuprammonium, Bemberg, and cellulose acetate (Celanese and American) silk yarns, are 207·4, 37·45%; 153·0, 65·5%; 173·9, 23·5%; 194, 51·5%; 156·1, 57·0%, respectively. Cellulose acetate silk is thus not superior to cellulose silks when in the wet state.

A. J. HALL.

Swelling properties of [cellulose] artificial silks [in water and caustic alkalis] and their use for identification. W. WELTZIEN (Textilber., 1926, 7, 338—343).—The moisture absorption curves of cellulose, egg albumin, and gelatin are similar and of S-shape. Cellulose silk yarn increases 3·0—4·5% in length when wetted, but regains its original length when afterwards dried. A definite parallelism exists between the extensions in length of various samples of viscose yarn (120 denier) under approximately their breaking strain (125 g.) and their increases in length when immersed in water; extensions of 7·5—9·5% and 8·5—10·5% in air of 55% relative humidity correspond to extensions in water of 3·0—4·0% and 4·0—4·5% respectively. The swelling action of caustic soda on viscose and cuprammonium silks (measured by the weight of solution absorbed) reaches maxima of 540% and 850% respectively with solutions of 10—12%, the swelling rapidly diminishing on either side of the maximum. The swelling is capable of determination with considerable accuracy except at the maximum when the caustic liquor exerts solvent action on the silk. Viscose silk treated with solutions of caustic soda of increasing concentration shows an initial extension in length of about 4% with caustic soda of 0—1%, the extension then decreasing to zero with caustic soda of 6—7%, and the length further diminishing to a minimum with caustic soda of concentration approximately equal to that capable of producing maximum swelling. Cellulose silks treated with caustic soda suffer no change or a decrease in length (according as the concentration of caustic soda used is less or greater than about 6%) when subsequently washed with water, but all the treated samples of silk decreased in length considerably (3—9%) when afterwards dried. The action of caustic alkalis on cellulose silk removes latent strains (produced in the silk during its manufacture (*e.g.*, coagulation during spinning) so that whereas the swelling effects produced by treating untreated cellulose silks with caustic soda and then water are not reversible, the changes produced by similar treatment of alkali-treated silks are reversible. Various cellulose silks may be distinguished from each other by their different contractions in length during immersion in water after treatment with caustic soda of 8—10%; Chardonnet silk contracts considerably more than viscose silk, whereas cuprammonium silk shows a small extension in length. Cellulose silk simultaneously swollen and stretched in caustic soda has less affinity than untreated silk for direct dyes. Stretched and unstretched cellulose silk yarns which dye to the same depth of shade develop different shades when similarly dyed together after treatment with caustic soda of a concentration which produces no change in length of the yarns.

A. J. HALL.

Comparison of the swelling in caustic soda of natural celluloses and artificial silk. W. WELTZIEN, GERHARDT, and TOBEL (Seide, 1926, 31, 132—140; Chem. Zentr., 1926, II., 512).—Raw and bleached cottons swell equally in solutions of

caustic soda. Cuprammonium silk and cotton form alkali-cellulose compounds having the same composition ($2C_6H_{10}O_5 : 1NaOH$) when treated with 10% (minimum) and 16% solutions of caustic soda respectively, and cuprammonium silk swells to a greater extent than cotton (swelling is measured by the increase in weight of the fibres when immersed in solutions of caustic soda), both of these facts being considered as due to the greater porosity of cuprammonium silk (see preceding abstract).

A. J. HALL.

Ripening of viscose solutions. J. D'ANS and A. JÄGER (Kunstseide, 1926, 8, 82—84; Chem. Zentr., 1926, II., 133—134).—Further investigations (cf. B., 1926, 532) indicate that the presence of sodium sulphate, carbonate, or sulphite accelerates considerably the coagulation of viscose solutions, sodium sulphite having the least effect. The ammonium chloride ripeness is only slightly affected by the presence of sodium sulphate or carbonate, while sodium sulphite retards the ripening. The presence of 0·05% of gelatin has no appreciable influence on the changes which occur in pure viscose solutions and the effect of even 1% is small. Tannin, glycerin, sucrose, and dextrose increase the ammonium chloride ripeness of viscose solutions except when the latter are impure. Potassium ferrocyanide has but a small influence on the ammonium chloride ripeness.

A. J. HALL.

Sulphite tissue paper as material for nitrocellulose for celluloid. K. ATSUKI and M. ISHIHARA (Cellulose Ind., Tokyo, 1926, 2, 175—179).—Tissue paper made from sulphite pulp purified by boiling with alkali and bleaching was compared with a cotton tissue paper as a raw material for nitration. The sulphite paper ocntained 92·7% of α-cellulose as against 98·8% for the cotton paper; the sulphite paper was also denser and stronger than the cotton paper. When nitrated under similar conditions, the relative viscosities of the nitrates in ether-alcohol were: for the sulphite paper 41·2 and for the cotton paper 52·0, while a purified cotton wool gave a nitrate of viscosity 609. The β- and γ-celluloses present in the sulphite paper are nitrated and remain in the nitrocellulose; the nitrates of these lower celluloses are soluble in ether-alcohol and camphor-alcohol giving solutions of low viscosity. Their presence in the film or celluloid modify the gel-structure in the direction of discontinuity and the tensile strength and elongation are decreased. They do not adversely affect stability. The tenacity of films made with optimum camphor contents were: sulphite tissue, 4·72 kg. per sq. cm.; cotton tissue, 6·18; purified cotton wool, 8·24. There is no fundamental obstacle to the use of suitably purified sulphite pulp for the manufacture of celluloid; the alleged low yields on an industrial scale can probably be easily overcome by improvements in the quality of the paper and in the nitrating plant.

J. F. BRIGGS.

See also A., Aug., 823, **Degradation of cellulcse to an anhydrotrisaccharide** (IRVINE and ROBERTSON). 824, **Action of oxygen on alkali-cellulose** (WELTZIEN and ZUM TOBEL); **Preparation of primary lignin** (FRIEDRICH and BRÜDD).

Vapour pressure and electrical conductivity of wood. HASSELBLATT.—See IX.

Manuring experiments with sewage. ZIELSTORFF, KELLER, and SPUHRMANN.—See XVI.

PATENTS.

Treatment of textile and other materials [wood] to protect them from attack by insects. H. MAXWELL-LEFROY, and GRAESSER-MONSANTO CHEMICAL WORKS, LTD. (E.P. 253,993, 24.3 and 14.5.25).—Wood and textile fibres are impregnated with polyhalogenated naphthalenes, such as di- and tri-chloronaphthalenes. Liquid monochloronaphthalene and p-dichlorobenzene are also highly toxic substances, but they slowly volatilise from the treated materials and are therefore unsatisfactory. Impregnation is effected by immersing the material in aqueous emulsions or solutions in volatile solvents or by spraying, the treated material being afterwards dried. For example, yarn or fabric is impregnated with a 5% solution of tri- or hexa-chloronaphthalene in benzene or with an emulsion containing 25 pts. of monochloronaphthalene, 25 pts. of trichloronaphthalene, 3 pts. of ammonium oleate, and a suitable quantity of water. A highly satisfactory impregnating mixture for timber contains 1 pt. of paraffin (sold as "burning oil, 300°"), 1 pt. of p-dichlorobenzene, and 1 pt. of hexachloronaphthalene. Trichloronaphthalene exerts a specific toxic effect on white ants and the death watch beetle.

A. J. HALL.

Production of film-like bands from cellulose solutions. WOLFF & Co., E. CZAPEK, and R. WEINGAND (E.P. 254,946, 11.11.25).—Cellulose films are prepared by passing a film of a solution of cellulose (*e.g.*, viscose), deposited upon a conveyor band, over and under two upper and lower rows of freely rotating guide rollers mounted within a vat containing a coagulating solution (*e.g.*, a 20% solution of sodium sulphate) and between two vertical electrodes so that a direct current flows through the conveyor band and the film thereon; the film is thereby successively subjected to the action of a liquor which is alkaline (adjacent to the cathode), neutral (between the electrodes), and acid (adjacent to the anode). The conveyor band consists of fabric impregnated with celluloid or bakelite or other material resistant to acids and alkalis, and the guiding rollers are preferably recessed so that they exert no deleterious pressure on the film. Dyes which are soluble in alkalis and insoluble in acids may be added to the coagulating solution for the purpose of producing coloured films, and the temperature of the bath may be controlled by adjusting the distance between the electrodes and the current passing. A film of 0·002 cm. thickness may be produced at the rate of 1500 cm. per hr.

A. J. HALL.

Manufacture of [hollow] threads, filaments, bands and the like from viscose. Courtaulds, Ltd., S. S. Napper, and H. D. Gardner (E.P. 253,953, 17.9.24).—Hollow threads are produced by spinning in an acid coagulating bath containing a zinc salt, a viscose solution containing substances capable of liberating a gas by reaction with the acid during coagulation. For example, a mixture of 1000 pts. of freshly prepared viscose (containing 8—10% of cellulose) and 50 pts. of sodium carbonate dissolved in 250 pts. of water is spun into an aqueous bath at 45° containing per 100 pts., 8 pts. of sulphuric acid, 5 pts. of zinc sulphate, 14 pts. of sodium sulphate, and 9 pts. of glucose, the resulting threads being hollow throughout the greater part of their length. In addition to a zinc salt the acid coagulating bath may contain sodium, ammonium, magnesium, iron, aluminium, or chromium salts.
A. J. Hall.

Manufacture of [hollow] threads, filaments, bands and the like from viscose. Courtaulds, Ltd., H. J. Hegan and F. Bayley (E.P. 253,954, 17.9.24).—Hollow viscose threads are produced by spinning into an acid coagulating bath a solution of viscose in which the proportion of caustic soda is as low as 3—5%. The acid bath or the viscose solution may contain added substances capable of liberating bubbles of a gas within the threads during coagulation. A suitable viscose solution is prepared by impregnating 100 pts. of air-dry cellulose with an 18% solution of caustic soda at 20° for 2 hrs., pressing out excess of caustic lye so that the resulting alkali-cellulose weighs 300 pts., allowing it to ripen for about 3 days, then treating the product with 37 pts. of carbon disulphide at 25° for 3 hrs., dissolving the resulting cellulose xanthate in 800 pts. of a 0·2% solution of caustic soda, and adding a solution containing 35 pts. of anhydrous sodium carbonate in 270 pts. of water (see preceding abstract). A. J. Hall.

Dissolving cellulose esters and cellulose ethers. E. Pfiffner and M. Ow-Eschingen (E.P. 231,161, 16.3.25. Conv., 24.3.24).—Wood oils, *i.e.*, the oils insoluble or sparingly soluble in water, which are obtained in the distillation of wood alcohol, or fractions of these oils, are used as solvents for cellulose esters and ethers. J. F. Briggs.

Purification of cellulose ethers. Soc. Chim. des Usines du Rhône (F.P. 603,791, 22.12.24).—A solution of a cellulose ether is treated with a strong acid and the cellulose ether is afterwards precipitated by the addition of a liquid which is miscible with the solvent, but in which the cellulose ether is insoluble. For example, a solution of ethylcellulose in acetic acid or alcohol is treated with hydrochloric or sulphuric acid respectively and then poured into water. A. J. Hall.

[Treatment of] fibres. [Manufacture of paper pulp.] A. Tingle (Can. P. 253,459, 7.11.24; cf. B., 1924, 783; 1925, 36).—Fibrous material is digested with milk of lime containing sulphur, then the lime removed by suitable treatment, and the product ground (disintegrated). A. J. Hall.

Manufacture of cellulose from resinous wood. F. F. Heyerdal, and A. S. Thunes Mck. Vaerksted (F.P. 601,757, 10.8.25. Conv., 7.7.25).—Cellulose material directly suitable for the manufacture of white paper is prepared by digesting wood with liquors containing magnesium bisulphite. A. J. Hall.

Treatment of cellulose with nitric acid. A.-G. für Anilin-Fabr. (F.P. 603,725, 28.9.25).—Cellulose is exposed to vapours of nitric acid mixed with air or inert gases. A. J. Hall.

Manufacture of fibrous material from straw. West Virginia Pulp & Paper Co., Assees. of V. Drewsen (Can. P. 252,217, 31.5.23).—Straw is digested for 5—7 hrs. at 170—180° with an aqueous solution of sodium sulphite until disintegration is complete. A. J. Hall.

Manufacture of cellulose from resinous woods. West Virginia Pulp & Paper Co., Assees. of V. Drewsen (Can. P. 252,218, 31.5.23).—Wood chips are heated for 6—10 hrs. under pressure with a liquor containing 45—55% (calc. on the weight of wood) of sodium sulphite, then steam is blown through the product, essential oils being recovered from the condensed vapours and wood pulp afterwards separated from the residual lye. A. J. Hall.

Sizing cellulose fibres [paper]. J. A. De Cew (U.S.P. 1,589,947, 22.6.26. Appl., 19.9.25).—The size and alum are added to the paper stock after the fibres of the stock have passed through the Jordan mill. J. F. Briggs.

Manufacture of solid, non-hygroscopic products from cellulose waste lyes. A.-G. für Anilin-Fabr., Assees. of K. Brodersen (G.P. 423,095, 11.3.24).—Mixtures of cellulose waste lyes and non-hygroscopic sulphonic acids from pitches and resins are evaporated to dryness. The products (cf. Rolland; B., 1925, 800) are water-soluble, non-hygroscopic, retain the technically important properties of the original cellulose lyes, and are suitable for use as restraining agents in dyeing. A satisfactory product is obtained by evaporating to dryness a mixture of a cellulose lye (containing about 50% of solids) and water-soluble sulphonic acids from naphthol pitch. A. J. Hall.

Fireproofing of textile fibres and fabrics and other porous or absorbent substances. T. J. I. Craig, Assr. to P. Spence & Sons, Ltd. (U.S.P. 1,593,752, 27.7.26. Appl., 2.11.25. Conv., 18.8.24).—See E.P. 244,503; B., 1926, 152.

Retting of flax, hemp, and other textile plants. G. Austerweil and L. Peufaillit (U.S.P. 1,593,788, 27.7.26. Appl., 3.7.25. Conv., 7.7.24).—See E.P. 236,590; B., 1925, 753.

Process of rendering chlorides of ketones [in cellulose pulp] soluble by treating with sulphites made from waste liquors. A. R. de

Vains (U.S.P. 1,593,487, 20.7.26. Appl., 19.10.23. Conv., 14.12.21).—See E.P. 198,975; B., 1923, 765 a.

Production of moistness-resisting papers. E. Fues (U.S.P. 1,593,296, 20.7.26. Appl., 21.10.22. Conv., 29.10.21).—See E.P. 187,987; B., 1924, 290.

Acidising [carbonising] textile fabrics and the like. W. M. R. Jahr (E.P. 255,725, 7.1.26).

Nozzles for the production of artificial filaments. L. A. Levy (E.P. 255,261, 14.9.25).

Drying or chemically treating continuous lengths of fabric or paper. A. Lambrette (E.P. 255,297, 2.12.25).

Tanning animal hides (G.P. 423,096).—See XV.

Chromium oxide compounds for tanning (G.P. 423,138).—See XV.

Ester mixtures (E.P. 250,910).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Catalytic action of iron and copper compounds in the bleaching of vegetable fibres with sodium perborate or perborate detergents. Y. Dalström (Svensk Kem. Tidskr., 1926, **38**, 96—101; Chem. Zentr., 1926, II., 506—507).—Contrary to the conclusions of Heermann (B., 1923, 306 a) it is concluded that the presence of iron and copper salts does not promote the deterioration of vegetable fibres during their treatment with solutions containing sodium perborate. In concentrated perborate solutions copper and copper plus iron salts are injurious, but iron salts alone are not harmful. The presence of the sodium salts of fatty acids is of special importance in this connexion. When they are present the copper and iron salts do not exhibit their catalytic properties. The bleaching action of a solution of sodium perborate containing a sodium salt of a fatty acid is greater than that of a similar pure solution of sodium perborate or one containing sodium carbonate. A. J. Hall.

Dyeing properties of wool after exposure to sunlight. W. von Bergen (Textilber., 1926, **7**, 451—457).—Thirty-four dyes representing all classes of wool dyes are classified as positive or negative according as they yield darker or lighter shades respectively on wool the chemical properties of which have been affected by sunlight as described earlier (B., 1926, 312). Positive dyes include Chrysoidine G extra, Anthracene Chrome Brown SWN, Diamond Fuchsin, Rhodamine 2B extra, Alizarin Saphirol SE, Methylene Blue BB, and Thioindigo Scarlet G, while Helianthin, Eriochrome Cyanin R, Alizarin Cyanine Green G extra, Indigo, and Thioindigo are negative. The positive or negative behaviour of a dye towards wool usually increases with an increase of the duration of exposure to light, but after a direct exposure of about 300 hrs. (equivalent to about 1800 hrs. under glass) the effect diminishes (as shown with Indigo and Anthracene Chrome Brown SWN); this limit coincides with the almost complete destruction of the epithelial scales of the wool and also with the maximum staining of the wool brown by means of tin salts (Engel's test). Indigo is positive towards wool after an exposure of 100 hrs., but is strongly negative after 264 hrs. The behaviour of acid dyes towards exposed wool varies according as they are applied from neutral or acid dye-baths, whereas indigo is negative whether applied from an alkaline vat or in the form of Indigosol O from an acid bath. Alizarin Cyanin Green E is negative when applied from a neutral bath or one containing 3% of acetic acid, but is strongly positive when applied from a bath acidified with 2% of sulphuric acid; Sulphocyanin GR extra (By.) behaves somewhat similarly. The differences in the behaviour of dyes towards exposed wool are very considerably diminished when the wool is chromed before dyeing. The intense yellow colour of exposed wool dyed by immersion in a cold aqueous solution of Methyl Orange is not removed by washing with water, whereas under similar conditions non-exposed wool is readily decolorised. As shown by treatment with hæmatin, the interior of exposed wool is alkaline (bluish-violet with hæmatin) and the outer part is acid (yellow-brown), the penetration of light being small. After prolonged exposure to sunlight wool becomes brittle and the fibres may be easily broken. Exposed wool is much more easily wetted-out than unexposed wool. A. J. Hall.

Physics and chemistry of dyeing processes. III. Dyeing wool with acid dyes. K. H. Meyer and H. Fikentscher (Textilber., 1926, **7**, 605—610).—Contrary to the view of Fort (B., 1913, 907) and confirming that of Knecht (B., 1904, 981), the dyeing of wool with acid dyes is considered to consist of salt formation due to direct chemical combination of the wool (base) with the dye (acid). As shown by quantitative measurement of the absorption of Orange I (*p*-sulphobenzeneazo-*α*-naphthol) in $N/80$ solution and a yellow dye (*p*-sulphobenzeneazoacetanilide) in $N/20$ solution by fine, medium, and coarse wool fibres the absorption is independent of the surface area of the fibres. Under conditions which produce no profound chemical changes in the wool substance, wool combines in constant proportions with various types of acids. For example, 100 g. of carefully purified wool (0·42% of ash) immersed for 5 hrs. (extended immersion had no appreciable influence on the result) at 50—60° in solutions (mostly $N/10$) of about 17 mineral, organic, and aromatic sulphonic acids, combined (within the limits of experimental error) with 0·08 g.-equivalent of the acid. The combination of wool with acids in solutions of high concentration is less definite since the wool substance is attacked and further basic groups are liberated. The combination of wool with acids is due to the presence in wool of about 1·1% of basic nitrogen (wool contains about 15% of nitrogen) of which about 0·4% is present in the form of free amino-groups. 100 g. of wool de-aminated by treatment with acetic acid and sodium nitrite combines with

about 0·048 g.-equivalent of an acid. Wool is capable of chemically combining with considerable quantities of dyes containing sulphonic acid groups, *e.g.*, wool immersed for 5 hrs. at 80—90° in a solution of the dye obtained by coupling tetrazotised dianisidine with dichlorobenzoyl-K-acid absorbed 24% of the dye. In many instances dyes form insoluble compounds with wool thereby affecting the fastness and evenness of the resulting shade. In dyeing processes it is seldom that more than 6—8% (calc. on the wool) of the acid dye is used, so that the residual basicity of the wool is saturated by the mineral or organic acid present in the dye-bath. For example, wool dyed with 2% of Wool Fast Red 4BA and 3% of sulphuric acid combines with 1·36% of the latter and of this acid 0·26% may be removed from the dyed wool by treatment for 2 hrs. at 90° with distilled water. A. J. Hall.

" Blinding " of viscose dyed with insoluble azo colours. F. M. Rowe (J. Soc. Dyers and Col., 1926, **42**, 207—209).—Viscose silk dyed in deep shades with insoluble azo dyes may become " blind " (non-lustrous) immediately or when subsequently soaped at temperatures exceeding 60°, the effect being particularly marked with certain combinations of naphthols and bases. For example, viscose dyed with the combination (orange) of 1% of Naphthol AS·RL (*p*-anisidide of *β*-hydroxynaphthoic acid) and 5% of Fast Orange G salt (stabilised diazotised *m*-chloroaniline), and heated at 60° with soap and soda is lustrous, but becomes bluish-red and " blind " when boiled. Similar effects are observed with combinations of Fast Orange G salt and Naphthol AS·BO (*α*-naphthalide of *β*-hydroxynaphthoic acid) or Naphthol AS·RL, but colours obtained with Naphthol AS·SW (*β*-naphthalide of *β*-hydroxynaphthoic acid) and Fast Red KB base (2-amino-4-chlorotoluene), and from Fast Black LB base (2-ethoxybenzene-azo-*α*-naphthylamine), Fast Black B salt, Fast Garnet G and GB bases (*o*-aminoazotoluene) are least likely to produce "blinding." " Blinding " is due to aggregation of the colouring matter within the viscose fibres, for while dyed " non-blinded " viscose silk appears to be evenly stained, " blinded " silk consists of a suspension of aggregates of the dye in lustrous colourless viscose. " Blinding " appears to be a function of the azo-dye and not of the diazotised base or naphthol and is related to the " blinding " of cellulose acetate silk by crystallisation of the dye within the fibres. A. J. Hall.

Adsorption of Methylene Blue by zinc oxide Burns and Wood.—See IV.

Patents.

Dyeing cellulose esters and ethers. W. Duisberg, W. Hentrich, C. Weinand, and L. Zeh, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,587,708, 8.6.26. Appl., 8.6.25. Conv. 26.6.24).—Aminoanthraquinone-*o*-sulphonic acids in which one of the hydrogen atoms in the same ring as the NH_2- and SO_3H-groups may be replaced by a halogen, a carbon radical, or an S-carbon radical, have a strong affinity for, and give fast shades on, cellulose esters, *e.g.*, the formate or acetate, and cellulose ethers, *e.g.*, ethylcellulose. The dyes are applied in aqueous solution, either directly or as the sodium salts, below 70°, 0·5—3 pts. of dye being used in 0·05—0·1% solution per 100 pts. of fibre. The simpler compounds yield yellow to orange shades, and substituted derivatives can be prepared to give a complete range of colours. 1-Bromo-4-amino-anthraquinone-3-sulphonic acid gives yellowish-red shades and 2-aminoanthraquinone-3-sulphonic acid yellow shades. T. S. Wheeler.

Dyeing, printing or stencilling of cellulose acetate. Brit. Celanese, Ltd., and G. H. Ellis (E.P. 253,978, 18.3.25).—Colouring matters of the stilbene group containing no sulpho-groups are suitable dyes for cellulose acetate, and they may be applied in a solubilised form (cf. E.P. 224,925; B., 1925, 39) or may be formed within cellulose acetate fibres by coupling diazotised aminostilbenes with suitable developers. Satisfactory dyes are stilbenedisazobisphenol (yellow), stilbenedisazo-*m*-toluidine (gold), stilbenedisazo-*α*-naphthylamine (red), aminostilbene-azo-*m*-phenylenediamine (gold), and aminostilbeneazo-*m*-toluidine (yellow). Diaminostilbene applied to cellulose acetate and then diazotised and coupled with phenol, 1-phenyl-3-methyl-5-pyrazolone, diethylaniline, *m*-phenylenediamine, *α*-naphthylamine, *β*-naphthol, resorcinol, or *β*-hydroxynaphthoic acid yields gold, red, red, reddish-brown, reddish-brown, plum, plum, or black shades respectively. A. J. Hall.

Dyeing cellulose esters. J. Y. Johnson. From Badische Anilin- & Soda-Fabr. (E.P. 251,155, 14.8.25).—See F.P. 600,106; B., 1926, 536.

Dyeing, colouring or coating of textiles and other articles [with atomised dyes]. Two-Tone Corp., Assees. of P. Mijer (E.P. 229,351, 16.2.25. Conv., 14.2.24).

Apparatus for treating yarn or other fibrous material [under tension] with a liquid. T. McConnell (E.P. 255,257, 9.9.25).

Apparatus for dyeing raw stock. G. W. Johnson. From Hussong Dyeing Machine Co. (E.P. 255,270, 23.9.25).

Catalytic reactions (G.P. 423,542).—See I.

Non-hygroscopic products from cellulose waste lyes (G.P. 423,095).—See V.

Mixtures yielding salts of sulphochloroamides (E.P. 241,580).—See XXIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Feasibility of coke-oven hydrogen for synthetic ammonia. B. F. Dodge (Chem. Met. Eng., 1926, **33**, 416—420).—The comparative costs of production of hydrogen by Claude's liquefaction process from coke-oven gas, and by the water-gas catalytic process, under American conditions, are calculated.

Cost of liquefaction per 1000 cub. ft. of coke-oven gas is assumed to be 12·6 c., this figure being based on various estimates of the cost of production of liquid oxygen and nitrogen. Data for the water-gas catalytic process are those of Patart relating to the Oppau plant, modified. If coke-oven gas can be bought at 10 c. per 1000 cub. ft. the final cost estimates for the two processes are almost identical. Cost of production by the Claude process might, however, be lowered by the simultaneous recovery of crude benzol and of ethylene for conversion into alcohol, or by the devising of some practical method of increasing the hydrogen content of coke-oven gas. C. Irwin.

Determination of total alkalinity in presence of alkaline-earths. R. Bürstenbinder (Chem.-Ztg., 1926, **50**, 516).—10 g. of the substance are extracted with warm water and the filtrate is titrated with hydrochloric acid, using methyl-orange as indicator. If the alkalinity of the filtrate is small, then alkaline-earths only are present. In the residue alkaline-earths are determined by the usual indirect method. S. K. Tweedy.

Equilibrium in the system: silica–alumina–calcium oxide–sodium oxide. W. Eitel (Z. Elektrochem., 1926, **32**, 336—341).—A review of recent work on the various binary and ternary systems involved, and a survey of the present state of our knowledge of the quaternary system. J. S. Carter.

See also A., Aug., 813, **Formation of mother-of-pearl** (Ganguly); **Preparation of silicon tetrachloride from silica** (Budnikov and Schilov).

Systems $MnO–Al_2O_3–SiO_2$, $MnS–MnSiO_3$, and $CaS–CaSiO_3$. Glaser.—See X.

Patents.

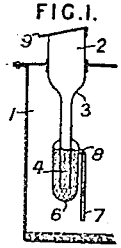
Concentration of dilute sulphuric acid. A. Grosche (G.P. 428,789, 5.4.24).—Dilute acid is treated with the final gases of the sulphur trioxide absorption apparatus of the contact process. W. G. Carey.

Production of gases resulting from the combustion of sulphur [for fumigation]. L. F. Pollain (E.P. 230,441, 26.2.25. Conv., 6.3.24).—A sulphur burner of hand-charging type is fitted with a primary air supply through a perforated tube and a preheated secondary air supply. After admission of the latter the gases pass through a superheater in contact with the burning sulphur, whereby combustion of the sulphur is completed and a considerable proportion of sulphuric anhydride is produced. The gases are then passed through a water cooler. The burner is intended for the production of toxic gas for fumigation purposes. C. Irwin.

Apparatus for burning sulphur. V. G. R. Allienne (E.P. 245,138, 22.12.25. Conv., 24.12.24).—A horizontal rotating cylinder, the upper part of which is enclosed by a fixed vessel to contain sulphur, has a device for dividing incoming air into two currents, one for the combustion of sulphur and the other for its final oxidation. The apparatus is started by melting sulphur inside the rotating cylinder; the sulphur distributes itself over the whole inner surface and exposes a large surface to the action of the air, while the heat of combustion of this initial charge melts the sulphur in the fixed vessel, and it is admitted to the rotating cylinder as necessary through needle valves. A fan is provided to draw air into the apparatus and serves also to draw the gases through a cooling chamber situated under the rotating cylinder. W. G. Carey.

Means for charging furnaces [with sulphur or other fusible material]. P. A. Ducatel (E.P. 249,547, 18.3.26. Conv., 20.3.25).*—Sulphur or the like is charged through a hopper, 2, having a lid, 9, and a lower tubular extension, 4, dipping below the surface of the molten material in a siphon trap, 6, 7, 8, within the furnace or burner. A liquid joint is thus formed and no gas or air leakage can occur. C. Irwin.

FIG. 1.



Obtaining by-products and heat from burning sulphur, pyrites, hydrogen sulphide, sulphurous acid, and other incompletely oxidised sulphur compounds. T. Chmura (F.P. 603,989, 15.7.25. Conv., 15.7.24).—Air containing incompletely oxidised sulphur compounds is treated so that the sulphur compound combines with the oxygen present to form sulphur dioxide and trioxide, which are separated from the nitrogen by compression to 1000 atm. and, if necessary, washing with suitable solvents. The nitrogen is then applied to the high-pressure synthesis of ammonia. A method of utilising the heat evolved when the sulphur compound is oxidised consists in passing the gas containing the sulphur compound and oxygen into a cylinder fitted with a piston and inducing combination by permitting ultraviolet rays to enter by means of a quartz window in the cylinder, admission being regulated by a rotating shutter coupled to the piston. A large number of methods of applying the invention are described. T. S. Wheeler.

Purifying and bleaching heavy spar. K. Ebers (E.P. 245,155, 24.12.25. Conv., 24.12.24).—Ground and roasted heavy spar is mixed with manganese dioxide and is added to hydrochloric acid and the whole heated. The chlorine evolved dissolves the iron oxide and other impurities and bleaches the product, the excess chlorine being absorbed in lime, and the excess acid being drawn off for further use. W. G. Carey.

Method of separating radium and barium salts. I. Bashiloff (E.P. 254,539, 31.7.25).—A cold concentrated aqueous solution of radium and

barium chlorides is subjected to repeated fractional crystallisation with the addition of suitable amounts of a salt of the same ion as the solution itself, and which does not form a soluble compound with the radium or barium salts. Such salts are calcium chloride or aluminium chloride, and the method is described in detail, using calcium chloride.

W. G. Carey.

Extracting radium from radium-barium salts and the like. H. Fleck, Assr. to W. A. J. Bell (U.S.P. 1,588,383, 8.6.26. Appl., 15.8.22).—A mixture of radium and barium sulphates, chlorides, or bromides (5 pts.) is dissolved in 40 pts. of a 50% aqueous solution of oxalic acid and after boiling for 30 min. the solution is filtered and diluted with 600 pts. of water. The product which separates on standing contains a greater proportion of radium salt than the original mixture. The precipitation is hastened by adding a small quantity of sulphuric acid, when the mixed sulphates separate.

T. S. Wheeler.

Process of producing alumina, alkali, and dicalcium silicate. A. H. Cowles, Assr. to Electric Smelting & Aluminum Co. (U.S.P. [a] 1,591,364 and [b] 1,591,365, 6.7.26. Appl., 17.5.20).—(a) A mixture of substances containing soda, lime, alumina, and silica is sintered to yield a product containing 1 mol. SiO_2 to 2 mols. CaO, and more than 1 and substantially less than 1·76 mols. Na_2O to 1 mol. Al_2O_3, and sodium aluminate is extracted from the product by leaching. (b) A mixture containing combined alumina and silica, calcium carbonate, and sodium carbonate is heated to a sintering temperature, the proportions being such that the furnaced product contains 1 mol. SiO_2 to 2 mols. CaO, and 1 mol. Al_2O_3 to 1 mol. Na_2O. Sodium aluminate is dissolved from the product by means of water containing sodium oxide so that the resulting solution contains materially less than 1·75 and more than 1·10 mols. Na_2O to 1 mol. Al_2O_3. The alumina is separated from the solution in known manner. H. Royal-Dawson.

Method of removing liquid from cyanides. C. B. Jacobs, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,591,630, 6.7.26. Appl., 24.2.21).—Mother liquor is removed from a mass of sodium cyanide obtained by melting crystallised sodium cyanide in its water of crystallisation by subjecting it to directly applied relatively high pressure, while it is maintained at a temperature above the transition point from the hydrate to anhydrous sodium cyanide.

H. Royal-Dawson.

Production of alkali cyanides. F. J. Metzger, Assr. to California Cyanide Co. (U.S.P. 1,591,720, 6.7.26. Appl., 15.7.24).—A combination of alkali metal compounds, having a lower melting point than any one of them alone, is mixed with carbon and subjected to a sufficiently high temperature to ensure the fluidity of the alkali compounds. Carbon impregnated with alkali compounds for use in the manufacture of alkali cyanide is thus obtained.

H. Royal-Dawson.

Process of making alkali xanthates. E. C. Missbach (U.S.P. 1,591,723, 6.7.26. Appl., 1.6.25).—A solution of an anhydrous monohydric alcohol in carbon disulphide is caused to react with dry alkali hydroxide, the hydroxide and the alcohol being used in molecular proportions. H. Royal-Dawson.

Evaporation of brines. R. W. Mumford, Assr. to Amer. Trona Corp. (U.S.P. 1,591,725, 6.7.26. Appl., 15.8.22).—Foaming of brine during concentration is prevented or reduced by carrying out the evaporation in the presence of a non-volatile emulsion colloid. H. Royal-Dawson.

Process of making arsenates of manganese. W. L. Tanner, Assr. to Grasselli Chemical Co. (U.S.P. 1,591,795, 6.7.26. Appl., 30.7.23. Renewed 12.11.25).—A mixture of arsenious oxide and a manganese compound capable of oxidising arsenious oxide is heated to the temperature of reaction in the presence of water. H. Royal-Dawson.

Producing arsenate of lime. F. Bidaud, Assr. to Soc. Chim. des Usines du Rhône (U.S.P. 1,591,958, 13.7.26. Appl., 15.1.26).—Calcium arsenite is fed continuously into a furnace, part of the charge is heated to a temperature high enough to initiate oxidation of the arsenite, and heating is discontinued as soon as reaction begins. A current of oxidising gases is passed through the moving mass, and the temperature is regulated so as to ensure complete oxidation of the arsenite to arsenate.

H. Royal-Dawson.

Preparation of copper arsenate or copper arsenite, or double compounds containing chlorine. I.-G. Farbenind. A.-G. (G.P. 428,239, 16.9.24).—Basic cupric chloride salts are decomposed by salts of arsenious acid or arsenic acid, or by the free acids, in the last case if necessary with the addition of free alkali. If a finely divided product is required substances may be added which cause fine dispersion. W. G. Carey.

Process of treating alunite. G. S. Tilley (U.S.P. 1,591,798, 6.7.26. Appl., 20.5.24).—Alunite is digested with sulphuric acid of such strength and amount, as to dissolve nearly all the iron and potassium present, leaving a residue of material containing alumina which is soluble in hot sulphuric acid. H. Royal-Dawson.

Production of [barium manganate in manufacture of] alkaline-earth permanganates. R. E. Wilson, L. W. Parsons, and S. L. Chrisholm, Assrs. to Sec. of War, U.S.A. (U.S.P. 1,592,480, 13.7.26. Appl., 17.7.22).—As a step in the manufacture of alkaline-earth permanganates, barium manganate is prepared by adding barium peroxide and manganese dioxide to a fused alkali hydroxide.

H. Royal-Dawson.

Method and means for burning gases [containing phosphorus]. H. F. Noyes and R. Weigel, Assrs. to Victor Chemical Works (U.S.P. 1,592,616, 13.7.26. Appl., 4.4.25).—Gases containing phosphorus and its compounds are

burned continuously at a relatively high and constant temperature, and the specific heat of the products of combustion is utilised selectively in heat-utilising units. H. ROYAL-DAWSON.

Extracting potassium and phosphorus from rocks. J. F. CULLEN and T. E. HARPER, Assrs. to U.S. SMELTING, REFINING & MINING CO. (U.S.P. 1,592,757, 13.7.26. Appl., 6.5.21).—The rocks are smelted under reducing conditions, and compounds of phosphorus and potassium are recovered from the gases produced. H. ROYAL-DAWSON.

Treatment of solutions containing ferric sulphate, titanium sulphate, and free sulphuric acid. RADIUM AND RARE EARTHS TREATMENT CO., Assees. of W. T. COOKE (Austral. P. 20,299, 30.10.24). —Solutions containing the sulphates of iron and titanium and free sulphuric acid are rendered suitable for the recovery of titanium by adding barium sulphide, barium carbonate, sodium hydroxide, and sodium sulphide to reduce the iron and neutralise the free sulphuric acid. T. S. WHEELER.

Separation of iron compounds from compounds or mixtures containing iron oxide. KÖNIGSBERGER ZELLSTOFF-FABR. & CHEM. WERKE KOHOLYT A.-G. (F.P. 604,048, 11.9.25. Conv., 18.9.24).—The mixture or compound is treated with chlorine below 700°, using for this purpose residual gases. It is advantageous to treat the product with hydrogen or other reducing agent. W. G. CAREY.

Manufacture of ammonia and ammonium compounds from calcium cyanamide. FABR. NAT. DE PROD. CHIM. ET D'EXPLOSIFS, ANC. ETABL. GHINIJONET & DELATTRE (Addn. 29,830, 24.11.24, to F.P. 587,803).—Steam mixed with air or nitrogen is passed over the porous residue from the process described in the chief patent (cf. E.P. 223,919; B., 1925, 670) at a temperature not below 400°. Ammonia is formed from the undecomposed cyanamide; the steam is catalytically decomposed by the porous mass, the hydrogen combining with the nitrogen present to form ammonia. The final residue, consisting of carbon, calcium carbonate, and some alkali carbonate is heated in nitrogen or air at a still higher temperature. Cyanogen compounds are formed, which are subsequently decomposed by steam. S. K. TWEEDY.

Production of high-pressure nitrogen for ammonia synthesis. FENO-GES. FÜR ENERGIEVERWERTUNG, Assees. of R. MEWES (G.P. 414,596, 15.5.24).—High-pressure nitrogen, taken from the heat exchanger of the separation column during the fractionation of liquid air under pressure into nitrogen and oxygen, is used for the synthesis of ammonia, either directly, or if necessary, after further compression. W. G. CAREY.

Production of sodium nitrate. I.-G. FARBENIND. A.-G., Assees. of R. GRIESSBACH, K. RÖHRE, and K. EYER (G.P. 428,137, 23.5.25).—Glauberite, $CaSO_4$, Na_2SO_4, in aqueous solution is decomposed by nitric acid or nitrates, especially calcium nitrate, at a temperature above 75°. W. G. CAREY.

Manufacture of silica gel. W. CARPMAEL. From CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 242,234, 17.8.25).—The soluble salts remaining after mixing water-glass with an acid and gelatinising, are only partially washed out, the final product having a salt content of about 2% of the dried gel. The adsorptive qualities are not impaired. W. G. CAREY.

Manufacture of strongly adsorbent silicic acid. I. G. FARBENIND. A.-G., Assees. of F. STÖWENER (G.P. 428,041, 21.6.24).—An appropriately produced silica jelly is freed from fluid by pressure, subjected to mechanical treatment, gradually dried, and washed. The material before or during the mechanical treatment may be mixed with metals or metal compounds; the metals or compounds are afterwards partly or wholly removed. W. G. CAREY.

Apparatus for the manufacture of lead oxide. H. WARING (E.P. 254,352, 30.1.25).—Lead oxide dust is prevented from escaping from the interior of ordinary oxide pots by substituting for the glands for the agitator shaft, a box fitted over the aperture through which the shaft passes, and driving back the dust which tends to escape by a blast of air or steam, or both, directed into the box. W. G. CAREY.

Manufacture of nitrides. H. JANISTYN (Austr. P. 103,216, 20.9.20).—Metals forming nitrides are deposited electrolytically, using a fluid metal cathode, and the alloy obtained is then subjected to the action of nitrogen at a high temperature. Hot solutions of alkaline-earth or lithium salts are used as electrolytes, and alloys melting below or about 100°, such as Wood's alloy, are used as cathode. W. G. CAREY.

Process for producing oxygen, either pure or mixed with other fluids. SOC. ANON. LE SALVOXYL (E.P. 252,319, 16.12.25. Conv., 23.5.25).—The anhydrous oxide of an alkali or alkaline-earth metal is added as a compact block to slightly acid hydrogen peroxide. The oxide is such that it will absorb the oxygen of the peroxide and form an unstable dioxide which will instantly decompose and allow the oxygen to be set free. Essences or other substances may be added to the peroxide or mixed with the blocks. W. G. CAREY.

Continuous production of sulphur from ammonium polysulphide solution. I.-G. FARBENIND. A.-G., Assees. of P. KOPPE and H. OEHLER (G.P. 428,087, 14.3.25).—The polysulphide is added to a fluid in a closed boiler maintained at ordinary or raised pressure, and at a temperature above the melting point of sulphur. A solution of a salt may be used which under the working conditions boils above the melting point of sulphur. W. G. CAREY.

Preparation of solutions of yellow arsenic. I.-G. FARBENIND. A.-G., Assees. of F. WINKLER (G.P. 428,040, 22.7.24).—The vapour of a solvent

for yellow arsenic is led over strongly heated arsenic and immediately condensed. W. G. CAREY.

Production of phosphoric acid. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of R. SUCHY (U.S.P. 1,593,514, 20.7.26. Appl., 15.12.25. Conv., 17.1.25).—See G.P. 423,275 ; B., 1926, 320.

Separating hafnium and zirconium. A. E. VAN ARKEL and J. H. DE BOER, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,582,860, 27.4.26. Appl., 5.8.24).—See E.P. 221,802 ; B., 1926, 156.

Catalytic reactions (G.P. 423,542).—See I.

Production of oxides and other compounds of zinc, lead, etc. (U.S.P. 1,591,253).—See X.

Electrolysis of alkali chloride solutions (E.P. 254,163).—See XI.

VIII.—GLASS; CERAMICS.

Factors governing the resistance of pots to glass attack. J. F. HYSLOP (J. Soc. Glass Tech., 1926, 10, 148—152).—In the case of two series of pots, K1 and K2, of which K1 had a longer average life in the commercial melting of a lead glass, it was found that the white layer adjacent to the glass was very uniform in the K1 series but quite irregular and considerably thicker in the K2 pots. *X*-Ray analysis of this layer showed the presence of mullite, cristobalite, and glass but with a higher silica content in the K1 series, indicating a more viscous layer with a slower rate of reaction between pot and glass. Both series of pots consisted of silicious grog in a clay matrix, but the alumina content of the matrix was higher in the K1 series. Squatting tests of cones of the clays in question, mixed with a standard glass, revealed the superior resistance of the matrix of the K1 pots, probably a decisive factor influencing the rate of pot attack. A. COUSEN.

Experimental tank blocks. W. A. MCINTYRE (J. Soc. Glass Tech., 1926, 10, 170—176 ; cf. B., 1926, 585).—In practical tests upon a series of tank blocks from fireclay or ball clay with silica and/or sillimanite, the mixture fireclay 25, silica 5, sillimanite (<10 mesh) 70 appeared most satisfactory when both manufacture and use were considered. By omitting the silica and suitably weathering, a porosity as low as 17·24% could be obtained. A. COUSEN.

Influence of moisture on the mixing of batches for soda–lime–silica glasses. M. PARKIN and W. E. S. TURNER (J. Soc. Glass Tech., 1926, 10, 114—128).—With normal sand–soda ash–limestone batches mixing is best accomplished with a moisture content of 3—4%, even when the particles vary considerably in size. Batches containing less than 1 or more than 6% of moisture are unsatisfactory for mixing. Less satisfactory results are obtained with fine soda ash and limespar than when material of normal grain size is used. Three types of "balling" occur, viz., (1) with sand acting as a nucleus, (2) soda ash caking, prominent only with light ash, (3) formation of limespar–soda ash pellets when wet limespar is used. Preferably, water, when required, should be added to the batch as a whole. Batches containing, in addition, saltcake or alumina give similar results. A. COUSEN.

Influence of water on the rate of melting and the working characteristics of soda–lime–silica glass. E. M. FIRTH, F. W. HODKIN, M. PARKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1926, 10, 129—145).—The melting of normal sand–soda ash–limespar and sand–soda ash–saltcake–limespar batches is, in general, beneficially affected by the presence of moisture in the batch, an optimum value being 1—2% in the former and 3—4% in the latter case. With a moisture content above a certain value, viz., 5% for the soda-ash batches, the glass obtained is more viscous and has a shorter working range than the glass from the dry batches. The saltcake batch gives a glass which fines more easily and is freer from wave than the batch with soda ash only. Differences in each series are not due to variations in chemical composition, and glasses from wet and dry batches have similar ignition losses either at temperatures up to 920° for 2 hrs., or at 330° and 2—3 mm. for 22 hrs. A. COUSEN.

Effects of chlorides on the melting and working properties of potash–lead oxide–silica glass. E. M. FIRTH, F. W. HODKIN, C. M. MUIRHEAD, M. PARKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1926, 10, 176—198).—When potassium chloride was added to normal lead crystal batches, the maximum amount retained in melting at 1350—1400° corresponded to 0·70—0·75% of chlorine, this being reached with 6 pts. per 100 of sand. With chloride not exceeding 2 pts. per 100 of sand practically the whole was retained in the glass. With large amounts, part decomposed to leave potash in the glass, and hydrochloric acid was evolved. Potassium chloride appeared either to diminish or render more uniform, volatilisation of red lead. Up to 2 pts. per 100 of sand, the chloride slightly retarded melting ; from 2 to 4 pts. the retardation was appreciable. Less than 2 pts. per 100 retarded, but larger amounts accelerated, the rate of fining. Dissolved chloride reduced waviness and improved the brightness of the glass, and although stirring failed to reduce wave, it assisted the escape of chloride. Moisture, varying from 2 to 4%, slightly assisted melting and fining, and up to 2·5% reduced wave, but in large amounts gave fine seed. The moisture appeared to assist decomposition of the chloride and caused a reduction of the proportion of the oxides of iron, aluminium, and manganese in the glass. Batches melted and fined more rapidly at 1400° than at 1350°, but waviness was greater, and seed recurred on keeping at 1150°. With increasing amounts of potassium chloride, the glass became increasingly green, although the proportion of iron oxide was apparently reduced. A. COUSEN.

Production of opalescence by chlorides in potash–lead oxide–silica glasses. E. M. FIRTH,

F. W. HODKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1926, 10, 199—212).—Chlorides are liable under certain melting conditions to produce opalescence in lead crystal glass either on melting or reheating. At least 2% of potassium chloride (or sufficient to leave 0·5% of chlorine in the glass) was needed in the batch melted at 1350° to give the effect over the range 1350—1150°, and harder glasses required more chloride. Below 500°, no opalescence was caused on reheating, the optimum value lying between 800° and 900°, and the rate of production of opalescence corresponded to the chloride present. The colour rapidly disappeared at 1000—1200°. Chloride apparently raised the annealing temperature of the glass. Suggested causes of opalescence are precipitation of either silica or lead chloride. A. COUSEN.

Devitrification of a lead borate glass. C. J. SMITHELLS (J. Soc. Glass Tech., 1926, 10, 145—146). —A lead borate glass in which a tungsten filament was embedded, on keeping at room temperature and then etching with alkaline potassium ferricyanide, showed devitrification effects after two years, and the whole glass appeared crystalline after 5 years. A. COUSEN.

Thuringian glass. F. ECKERT (Glastech. Ber., 1926, 3, 435—444).—The reputation of Thuringian glass is due to its ease of working and stability before the lamp, and these result from the presence of alumina (introduced by the use of felspathic sand), and a high general alkali content with relatively much potash. The firm of Schott & Gen. has improved the chemical resistance of the glass by reducing the alkali, increasing the alumina, and adding boric oxide. Weber's discovery of the effect of the simultaneous presence of two alkali oxides in promoting thermal afterworking resulted in the production of "Normal" thermometer glasses 16 III and 59 III. The average percentage compositions of the three chief types of Thuringian glass are:—(1) apparatus glass: SiO_2 68·5, Al_2O_3 3·2, Na_2O 14·2, K_2O 6·3, CaO 7·1; (2) packing and medical glass: SiO_2 68·7, Al_2O_3 3·9, Na_2O 17, K_2O 3·8, CaO 7·4; Christmas ware: SiO_2 68, Al_2O_3 3·1, Na_2O 22, K_2O a little, CaO 7·2; all contain small amounts of As_2O_3, Mn_2O_3, NiO_2, and Fe_2O_3. Examination of glasses from some 30 factories indicated that 4/5 were of low chemical stability and fell in group V of the Reichsanstalt classification. None of the glasses of types (2) and (3) reached group IV and of type (1) only 1 sample fell in the soft apparatus and 1 in the hard apparatus class. Where difficulty of joining samples of glass occurs, it is due to a difference of past heat treatment rather than to difference of composition. Appearance of colour on heating may be caused by the presence of the oxides of arsenic, antimony, zinc, or iron in the glass. Roughening of the glass in the flame has been attributed to divitrification and to volatilisation of alkali, but it may be produced by colloidal separation. The phenomenon is hindered by replacement of silica by alumina, by basic earths, or, other things permitting, by alkali. The presence of potash tends to prevent roughening, whilst it also causes an improvement in working qualities. Alumina is likewise indispensable. A. COUSEN.

Physico-chemical investigation of Borowltz refractory clay. G. G. URASOV (Z. anorg. Chem., 1926, 154, 152—169).—The Borowitz clays vary in ceramic properties from "biscuit" with very little plasticity to highly plastic soft porcelain. All these clays when heated show halts at 110° and at about 600°, corresponding with loss of free and combined water, and a further sudden rise of temperature at 950° due to an exothermic reaction. The halt at 600° is less abrupt for the more plastic clays, corresponding with a more gradual loss of water, and the irregularity at 950° is also less pronounced. When a current of dry air is passed over the clays, heated at a constant temperature, the loss of water from the least plastic clays is about 3% at 110° and 24% at 387°, and from the plastic clays 13—22% at 110° and 47% at 387°. A large proportion of the water is evolved at about 400° and at 425° over 90% has been lost from both classes of clays. The higher temperature indicated by the heating curves for the loss of combined water is due to the systems not being in equilibrium when heated comparatively rapidly. If the combined water was water of crystallisation the whole would be lost at the same temperature, whereas the loss from colloidal substances is gradual. The loss from the non-plastic clay corresponds fairly closely with the former and that from the plastic clay more with the latter. The following scheme is proposed to represent the loss of water in three stages: 100—400°, α-$Al_2Si_2O_7,nH_2O \rightarrow \alpha$-$Al_2Si_2O_7,xH_2O + (n-x)H_2O$; 400°, α-$Al_2Si_2O_7,n_1H_2O \rightarrow \beta$-$Al_2Si_2O_7,mH_2O + (n_1-m)H_2O$; 400—600°, $\rightarrow \beta$-$Al_2Si_2O_7,yH_2O + (m-y)H_2O$; at 600° y is zero. The first and last systems are univariant and equilibrium is possible over a range of temperatures; the second system is non-variant. X-Ray examination of the clays showed no marked differences in structure. A. GEAKE.

Physico-chemical problems of the glass and ceramic industries. G. KEPPELER (Z. Elektrochem., 1926, 32, 378—382).

System silica–alumina–calcium oxide–sodium oxide (EITEL).—See VII.

PATENTS.

Manufacture of glass. GEN. ELECTRIC CO., LTD., Assees. of PATENT TREUHAND GES. FÜR ELEKTR. GLÜHLAMPEN M.B.H. (E.P. [A] 254,174 and [B] 254,175, 24.11.25. Conv., 1.8.25).—(A) Glasses for electrical insulating purposes, containing silica, potassium and sodium oxides, and the oxide of at least one bi- or tervalent metal, are prepared with a ratio of potassium oxide to sodium oxide lying between 7 : 1 and 3 : 1. Such relationship gives a minimum electrical conductivity, the optimum ratio being influenced somewhat by the remaining constituents of the glass. (B) Electrically insulating

glasses, as for incandescence lamps, containing silica and the oxides of potassium, sodium, and lead, are prepared with, in addition, a small proportion (but not less than 2%) of calcium oxide. By this means the electrical conductivity is reduced without raising the softening temperature excessively.

A. Cousen.

Unsplinterable glass and other vitreous cemented composite bodies. E. Hope (E.P. 254,668, 26.1.25).—Itaconic acid methyl or ethyl ester is heated to form a viscous liquid, which is used as a cement in the production of unsplinterable glass and like products. By further heating the polymerisation of the itaconic ester is completed. A small quantity of free itaconic acid is preferably added to the ester to act as a catalyst. A. Cousen.

Manufacture of a glass. Sibor S. A., Verreries de Romont (Swiss P. 114,242, 21.12.24).—A glass, the resistance of which to sudden temperature changes and to the action of chemical reagents is equal to that of a good porcelain, is made by melting together sand, borax, and aluminium sulphate.

A. R. Powell.

Brick kilns. J. Procter (E.P. 254,886, 24.7.25). —A continuous brick kiln is provided with an independent, external flue and with an exhaust fan or chimney. A number of tubular branches, which, by provision of suitable dampers allow of disconnexion from the flue or of connexion with the flue and the burning chamber, serve to control the air or gas passing through the kiln. Suitable covers are provided for sealing the passages to the kiln when the tubular members are removed. A. Cousen.

Furnace for glass manufacture. P. Arbeit, Assr. to Manufactures des Glaces & Prod. Chim. de St. Gobain, Chauny & Cirey (U.S.P. 1,593,054, 20.7.26. Appl., 19.11.24. Conv., 6.12.23). —See E.P. 225,837 ; B., 1925, 881.

Burning ceramic wares and apparatus therefor. E. C. R. Marks, From Ac Spark Plug Co. (E.P. 255,131, 16.3.25).

Decorating earthenware and like articles. W. Bourne and A. E. Hewitt (E.P. 255,336, 25.1.26).

Saggers and setters employed for firing pottery and other ware. F. Bennion and J. B. Clarke (E.P. 255,798, 15.6.26).

Electrically insulating coating on metals (F.P. 600,774).—See X.

IX.—BUILDING MATERIALS.

Constitution of cement. E. Jänecke (Z. Elektrochem., 1926, **32**, 354—362).—A review of the present state of our knowledge of the constitution of cement clinker. The system $CaO–Al_2O_3–SiO_2$ in so far as mixtures rich in lime are concerned, is discussed. Recent work confirms the existence of the compound $8CaO, Al_2O_3, 2SiO_2$ previously described by the author (B., 1912, 74, 387 ; 1915, 81) and which was considered by Rankin and Wright (B., 1912, 491) to be non-existent. Cement clinker contains dicalcium silicate (belite), tricalcium silicate, tricalcium aluminate, the compounds, $5CaO,3Al_2O_3$, $8CaO,Al_2O_3,2SiO_2$ (alite), $2CaO,Fe_2O_3$ (celite), and free lime. The so-called aluminous cements contain gehlenite, $2CaO,Al_2O_3,SiO_2$. J. S. Carter.

Permeability of Portland cement concrete. W. H. Glanville (Dep. Sci. Ind. Res., Building Res., Tech. Paper 3, 1926, 1—50).—The permeability of Portland cement concrete, as measured by the rate of flow of water through the concrete under a standard pressure, is determined chiefly by the amounts of water and cement used in mixing, under normal conditions of curing. For any given grading of cement and aggregate, the quantity of water which produces minimum volume of concrete also gives minimum permeability and maximum crushing strength. The influence of water content is greater with lean mixes, and more pronounced in the early stages of maturing. Cement and water content are of approximately equal importance. The proper grading of the aggregates affects the ease of working of the mix, *i.e.*, the proportion of water which must be used and therefore the permeability. The addition of finely powdered inert materials (*e.g.*, hydrated lime, slate waste) frequently lowers the permeability. Curing conditions are probably the most important factor in determining permeability, curing in water giving the best results. B. W. Clarke.

Water vapour pressure and electrical conductivity of wood in relation to its water content. M. Hasselblatt (Z. anorg. Chem., 1926, **154**, 375—385).—The water content of birch-wood was determined at relative humidities of 0—100% at the ordinary temperature. Very concordant results could not be obtained, probably on account of variations in the samples of wood. Usually the equilibrium was approached from the moist side, but results obtained from the dry side were the same within the experimental error. The moisture content of wood is considerably greater than that of pure cellulose, and at 40, 60, and 80% relative humidity is approximately 7·5, 12·5, and 20%, respectively. It is calculated that to squeeze out the water a pressure of 870 atm. is necessary at 50% relative humidity, and greater pressures when the wood is drier. In water dry wood swells 13·6% ; in organic liquids the swelling is less, being 9·5% in alcohol and less than 1% in hydrocarbons. Electrical conductivity was measured with thin pieces of veneer, in the direction of the radial marks. When the water content diminishes from its maximum to 7% the conductivity falls from 2×10^{-6} to 10^{-12}, and when the log. of the conductivity is plotted against the moisture content, a curve similar to the vapour pressure curve is obtained. By measuring the conductivity the moisture content is rapidly determined, with an error of about 0·5% when it is below 15%. A. Geake.

Utilisation of phenoxides from shale oil for wood preservation. N. Weiderpass and P.

KOGERMAN (Sitzungsber. Naturforsch.-Ges. Univ. Tartu. 1926, **33**, [1]. Reprint, pp. 1—12).—Esthonian shale oil does not possess so strong an antiseptic power as coal tar oil products, but the phenols and phenoxides prepared from the shale oil give much better results, the phenoxides being especially suitable for wood preservation. Phenoxides prepared from the refined oil are more effective than those from the crude oil. More prolonged tests are necessary before the durability of wood treated with these compounds can be definitely guaranteed.
B. W. CLARKE.

System silica-alumina-calcium oxide-sodium oxide (EITEL).—See VII.

PATENT.

Production of a matte surface on articles comprising a binder consisting of a heavy hydrocarbon compound. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of W. R. WHITNEY (E.P. 234,813, 26.5.25. Conv., 28.5.24).—The surface of articles, *e.g.*, switchboard panels, made from asbestos board containing a heavy hydrocarbon compound, such as asphalt or bitumen as a binding agent, is treated with a concentrated acid, such as sulphuric acid, thereby producing a matt surface, which has a more pleasant appearance than the normal shiny surface. B. W. CLARKE.

Bituminous emulsions. F. LEVY (E.P. 254,004, 25.3.25).—Rubber latex, in solution or as an aqueous suspension, is added in the proportion of 5—20% by weight, to molten or liquid bitumen during the process of emulsifying the latter, with, *e.g.*, oleic acid and caustic soda, and before the incorporation of the main body of the water with the emulsion. The product may be used as a preservative coating or as a binding agent, which can be applied without the aid of heat. If the emulsion is broken down, *e.g.*, by the addition of an electrolyte, the coagulum may be moulded to any desired shape, forming articles with a certain degree of resiliency. B. W. CLARKE.

Stable aqueous [bituminous] emulsions. C. A. BRAUN and G. S. HAY (E.P. 254,012, 25.3.25).—A stable aqueous bituminous emulsion can be prepared in unlimited quantities by mixing the material to be emulsified with alkaline water in presence of a small quantity of an emulsion of the material prepared previously by aid of an emulsifying agent such as gum arabic etc. B. W. CLARKE.

Cement and process of making it. H. KÜHL, Assr. to A. T. OTTO & SONS (U.S.P. 1,594,178, 27.7.26. Appl., 19.2.25. Conv., 31.3.24).—See E.P. 231,535; B., 1925, 923.

Treatment of wood to protect it from attack by insects (E.P. 253,993).—See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of the blast-furnace temperature on the properties of pig-iron. A. WAGNER (Stahl u. Eisen, 1926, **46**, 1005—1012).—In order to determine the cause of the difference in the physical properties of different samples of pig-iron and cast-iron of the same chemical composition, a series of tests were made in which the temperature of the blast was varied between 500° and 900°. The resulting iron was examined chemically, physically, and microscopically, but no basic differences in any of the properties were found that could be traced to differences in the temperature of the blast. Certain regular variations in the proportion of carbon present as graphite were noticed in the samples produced by the use of a highly heated blast, but these were found to be dependent on the relatively large fall in temperature during the period of blowing and not on the actual temperature of the furnace (cf. Pivovarski, B., 1926, 91, and Wedemeyer, B., 1926, 544). These apparently contradictory results are ascribed to the influence of the heat capacity of the slag in the blast furnace and it is shown that the iron produced has better mechanical properties the greater the amount of slag formed at the same time. The period of contact with the slag and the viscosity of the slag also appear to influence the properties of the metal obtained.
A. R. POWELL.

Oxidation of metallic iron and the pyrophoric properties of blast-furnace flue-dust. K. HOFMANN (Stahl u. Eisen, 1926, **46**, 916—918).—In the oxidation of iron, a layer of ferric oxide is first formed on the surface; the adjacent iron then acts as a reducing agent, causing a graduation from ferric oxide through a zone of magnetic iron oxide and ferrous oxide to an underlying layer of metallic iron. The term "reaction diffusion" is applied to this process. In the oxidation of an iron powder, complete conversion to ferric oxide occurs if the grain diameter is less than a certain size. Above that size, "reaction diffusion" occurs leading to the formation of magnetic iron oxide, more or less free iron remaining in the centre according to the diameter of the particles. The finer the grain size, and therefore the greater the free surface area, the higher the proportion of ferric oxide in the product. Iron containing finely disseminated ferric oxide gave a pyrophoric product after a short reduction in hydrogen below 600°. Exothermic absorption of oxygen on the surface of this material in air causes a temperature rise proportional to the ratio surface : mass. Spontaneous combustion takes place if this ratio is sufficiently large. The pyrophoric property is removed by heating for a long time, or at a high temperature in an inert atmosphere. L. M. CLARK.

Thermal effects of the iron-carbon eutectoid. R. RUER (Stahl u. Eisen, 1926, **46**, 918—920).—A reply to Hayes, Flanders, and Moore (Trans. Amer. Soc. Steel Treat., 1924, **5**, 183) on the decomposition of austenite, cooling in accordance with the stable system, to give iron and carbon with the formation of a eutectoid corresponding to pearlite in the metastable system. Immediately after solidification, a pure iron-carbon alloy with 3·85% of carbon, consists

of graphite and austenite. In the first cooling the transformations proceed according to the metastable system, the carbon separating from the austenite as cementite, and the residual austenite yielding pearlite and giving the Ac1 (metastable) arrest at 734°. On subsequent repeated heating and cooling, the pearlite arrest point becomes smaller and a new point Ac1 (stable) appears, due to the graphite+ferrite⟶austenite transformation, the carbon having been thrown out of solution on cooling as graphite or temper carbon. Ac1 (stable) occurs at 746° and is therefore 12° higher than Ac1 (metastable), these being the upper limits of the equilibrium values A1 (stable) and A1 (metastable). As the stable and metastable points both arise from mutual solution of two phases, Ac1 is probably higher than A1 by the same amount in both systems. Since A1 (metastable) is known to be 721°, A1 (stable) must be 733°. The criticisms by Hayes (*loc. cit.*) of Ruer's earlier work (B., 1921, 627 A) on account of the rate of heating and cooling and the maximum temperature employed are answered. Extrapolating to a zero rate of heating, Hayes' results became 759° and 771° for A1 (metastable) and A1 (stable) respectively—the difference of 12° between the values agreeing exactly with that found in the present work. The difference of 38° between the absolute values of A1 found in the two researches is ascribed to the excessive impurities (2·05%, including 0·95% of silicon) in the iron used by Hayes. L. M. Clark.

Origin of quenching cracks [in steel]. H. Scott (Sci. Papers U.S. Bur. Standards, 1925, 20, [513], 399—444).—The cracking of cylinders of hard tool steel when quenched was in all cases caused by tangential stress which was tensional at the surface of the specimen. Cracks rarely occur when the surface is under compression as the compressional stress that the steel will stand at the surface is very high as compared with the tensional stress. In practice a condition which produces cracks can be avoided by using a medium which will quench much more rapidly, or by retarded cooling (hot oil) during the hardening transformation, the treatment being determined by the shape of the piece and the composition of the steel. C. A. King.

Strengthening and recrystallisation of hardened steels. E. Houdremont, H. Kallen, and K. Thomsen (Stahl u. Eisen, 1926, **46**, 973—976).—If σ_0 is the tensile strength of an unworked steel and σ is its tensile strength after being drawn down from a cross-sectional area of F_0 to one of F, then $\sigma=\sigma_0+a \log F_0/F$, where a is a constant. When σ is plotted against $\log F_0/F$ a straight line is obtained which is steeper the harder the original steel. The angle of slope of the curve for hardened steel is practically the same as that for the same steel after annealing unless the hardening has been carried to an extreme. Subsequent annealing of the cold-drawn steel has little effect on the tensile strength below 300°; above 400° the tensile strength falls rapidly with the annealing temperature and at 550° reaches that of the steel after hardening, but before drawing, although no change can be seen in the microstructure. The effect of annealing below 550°, therefore, is simply to relieve the internal strain induced in the individual crystals by cold drawing. At higher temperatures recrystallisation commences and the tensile strength falls further while the pearlite collects into granular aggregates. All steels behave similarly under the above tests and none of the usual alloying elements appear to have any effect on this behaviour. A. R. Powell.

System iron–oxygen. K. Schönert (Z. anorg. Chem., 1926, **154**, 220—225).—The solubility of oxygen, as ferrous oxide, in α-, β-, and γ-iron is probably 0·03, 0·13, and 0·15—0·19%, respectively, increasing in γ-iron with rising temperature. In molten iron the solubility is 0·21% at the f.p., which is depressed to 1509°. When more oxygen is added two solid phases are formed, the oxygen-rich phase containing about 21% of oxygen and consisting of ferrous oxide with a small quantity of ferric oxide and 0·99% of free iron. From these data a portion of the equilibrium diagram is constructed. A. Geake.

Etching colours of alloys of iron and silicon. F. Körber (Z. anorg. Chem., 1926, **154**, 267—274).—When alloys of iron and silicon are etched with alkaline sodium picrate at 95° the thickness, y, of the film formed, as determined by its colour, is given by the equation $dy/dt=e^{-by}/ab$, where a and b are constants. The constant b is independent of the silicon content of the alloy, but increases with rising temperature; the constant a is independent of the temperature, but diminishes with increasing silicon content up to 3%. When the alloys are heated at 400° in air similar coloured films are formed, and their rate of formation changes more rapidly with changing silicon content. The film formation with alkaline sodium picrate may be used to detect, and to determine approximately, silicon in iron, provided aluminium and tin, which give similar films, are absent. A. Geake.

Surface improvement of metals by diffusion. I. Diffusion of chromium into iron and the limits of resistance of mixed crystals of chromium and iron. G. Grube and W. von Fleischbein (Z. anorg. Chem., 1926, **154**, 314—332).—When a rod of iron is surrounded with powdered chromium and heated in hydrogen the chromium content of the surface layer of the iron and the depth of penetration of the chromium increase with rising temperature and increasing duration of heating. Thus, after 24 hrs. at 1100° the surface layer contains 5·3% Cr, and the depth of penetration is 0·3 mm., and after 72 hrs. at 1280° the corresponding figures are 28·4% and 1·7 mm. After 96 hrs. at 1200°, 1280°, and 1320° the surface layers contain 43·3, 47·4, and 52·9% Cr, respectively; the composition is then constant for a depth of about 0·3 mm. and probably represents the solubility of chromium in iron at the several temperatures. The chromium content of a rod does not diminish uniformly with increasing depth, and the diffusion coefficient is

therefore different at different depths. The diffusion layer consists of mixed crystals of iron and chromium arranged radially. The rate of dissolution in dilute nitric acid diminishes with increasing chromium content; when this reaches 12·5 mol. % the rate of dissolution of chromium diminishes suddenly, and when it reaches 25 mol. % falls suddenly to zero, and the rate of dissolution of iron also shows a sudden drop to a low value. The iron dissolves more rapidly than the chromium relatively to its proportion in the surface layer. The protective effect of small proportions of chromium on the iron may be due to the dissolved chromic acid rendering the iron passive. The resistance of a bar is not diminished by rolling to half its initial diameter. A. GEAKE.

Etching figures on copper as a means of determining the crystal orientation. W. KÖSTER (Z. Metallk., 1926, 18, 219—220).—Electrolytic copper deposits have a fibrous structure in which the crystals are irregularly oriented about the [001] axis which is always in the direction of the current flow. Hence a section perpendicular to the cathode will show, on deep etching, figures characteristic of cube, octahedron, and rhombic dodecahedron faces, while a section parallel to the cathode exhibits figures characteristic of the last-named face only. The etching figures on cube faces are squares with one of the diagonals in the direction of current flow, those on the rhombic dodecahedron faces wavy lines parallel to the longer diagonal of the rhombus face, and those on the octahedron faces minute equilateral triangles, one side of each triangle being parallel to the direction of current flow. Annealing at 600—700° for 7 hrs. has practically no effect on the crystal orientation of electrolytic copper (cf. B., 1926, 546). A. R. POWELL.

Segregation phenomena in copper-zinc alloys. W. CLAUS (Z. Metallk., 1926, 18, 228—230).—Extremely rapid cooling of copper-zinc alloys containing 40—50% Cu, *e.g.*, by pouring into ice-cold water, results in the ordinary intercrystalline segregation according to the constitution diagram of these alloys; chill-cast alloys containing 15% Cu, however, exhibit inverse segregation. Brasses containing 60—70% Cu do not segregate at all under normal conditions; addition of a third constituent, *e.g.*, aluminium, antimonial lead, or phosphor-copper containing tin, causes segregation of a white constituent towards the walls of the mould, whether iron or sand, but no segregation occurs if the alloy is poured directly into water. It, therefore, appears that a certain predetermined rate of cooling is essential before inverse segregation takes place and this is in complete agreement with Masing's theory (B., 1925, 994). A. R. POWELL.

Technological behaviour of pressed brass bars. W. KÖSTER (Z. anorg. Chem., 1926, 154, 197—208).—The structure of a pressed brass bar changes along its length; at the beginning it is granular, the grains being β-mixed crystals with needle-shaped α-mixed crystals; at the end a section appears more uniform and consists of fine grains of α- and β-mixed crystals. The mechanical properties vary correspondingly, the fracture changing from granular to a very fine structure, and the elastic limit, tensile strength, and hardness increasing. This change is due to the cooling of the press block which is at first heated to 800°, and thus is in the range of β-mixed crystals. The change in structure along a bar depends on the conditions governing the cooling of the press block and also on the copper content of the brass. Subsequent heating or cold-working diminishes the differences between the ends of the bar but does not produce a completely uniform material. The end of the bar is more liable to crack than the beginning and this tendency is somewhat increased if it is heated at 600°. The temperature to which it is necessary to heat to avoid danger of cracking is greater for the end than for the beginning of the bar and it is advisable always to heat to about 200°. This does not affect the mechanical properties. A. GEAKE.

Expansion of iron-containing aluminium on solidification. G. MASING and O. DAHL (Z. anorg. Chem., 1926, 154, 189—197).—The eutectic mixture of aluminium and the compound $FeAl_3$ contains 2—3% Fe, both by calculation from the depression of the m.p. of aluminium, and by microscopical observation. The formation of warts on solidification of aluminium is due to the presence of iron, and is not affected by the silicon content. Their formation depends greatly on the rate of cooling. After melting in hydrogen wart formation was pronounced, in nitrogen it was almost absent, and it could not be observed when aluminium containing 13% of iron was fused in a vacuum. It is due to the liberation of gas, and metal containing warts has a much lower density than that which is free from them. A. GEAKE.

Influence of the heat treatment of aluminium on its rate of dissolution in hydrochloric acid. W. WIEDERHOLT (Z. anorg. Chem., 1926, 154, 226—237).—The rate of dissolution of aluminium in hydrochloric acid probably depends on the ease with which local electrical cells can be formed, and thus on the extent to which the impurities, chiefly silicon, are separated from the mass of the metal. Dissolution is most rapid when the aluminium has been heated to 300°; at this temperature equilibrium is attained, and the maximum separation of silicon occurs. At lower temperatures diffusion of the silicon is slower and less separation takes place; at higher temperatures the silicon diffuses into the aluminium with the formation of mixed crystals. When heated at a temperature below 300° the rate of dissolution increases with the duration of heating, and the rate of cooling is without influence. For temperatures above 300° the rate of dissolution decreases when the duration of heating is increased, and when the metal is subsequently cooled rapidly. A. GEAKE.

Rate of dissolution of aluminium in hydrochloric acid. M. WERNER (Z. anorg. Chem. 1926, 154, 275—293).—The rate of dissolution of a sample

of sheet aluminium containing 0·32% Si, 0·05% Fe, and traces of copper, zinc, and magnesium was determined after various heat treatments by measuring the volume of hydrogen evolved. The velocity fluctuates, being alternately faster and slower, and this is ascribed to the greater or smaller number of crystals of impurities which are exposed on the surface from time to time. In comparing different samples the velocities during the first period of rapid attack were used. The stability of aluminium towards acid is improved by heating and quenching; the temperature should not reach 500°. When aluminium which has been quenched from 400° is tempered, the rates of reaction for the first rapid period and for the succeeding slow period are increased owing to the decomposition of mixed crystals. The rate of decomposition is lower at 200° than at 300°, and is negligible at 100°. The Mylius test is improved by using 25 c.c. of *N*-hydrochloric acid instead of 5 c.c., the latter volume being insufficient to utilise fully the first period ot rapid action. The weight of aluminium dissolved by hydrochloric acid is greater than corresponds with the normal chloride, owing to the formation of basic chlorides. A. Geake.

System aluminium–zinc. O. Tiedemann (Z. Metallk., 1926, 18, 221—223; cf. B., 1926, 160).—Experiments on the effect of prolonged annealing, at various temperatures, of zinc–aluminium alloys containing up to 15% Zn have substantiated the division of the solid solution field into four parts. The tensile strength of alloys annealed at 500° for 2 hrs. and slowly cooled increases regularly with the zinc content from 5 kg./mm.^{2} with 0% Zn to 20 kg./mm.^{2} with 15% Zn, whilst the elongation decreases from 41·5% to 24% respectively. After annealing for 10 days at 240° and quenching, the tensile strength curve is practically identical with the foregoing, but the elongation increases with the zinc content from 21% with 0% Zn to 31% with 10% Zn, which is identical with the elongation of the corresponding alloy annealed at 500°, and the further course of the curve also follows that of the alloys annealed at 500°. The tensile strength of an alloy annealed at 350° for 7 days, quenched, and annealed at 240° for 10 days is less than that of the corresponding alloy annealed at 240° for 10 days when the zinc content is below 5%, the same when the alloy contains 5% Zn, and greater when more zinc is present; the ductility curve of the alloys after the former heat treatment resembles that obtained by annealing at 500°, but the ductility of any alloy annealed at 350° is less than that of the corresponding alloy annealed at 500°. The second annealing at 240° of alloys already annealed at 350° decreases the tensile strength when more than 7% Zn is present to an extent which is roughly proportional to the zinc content above 7%; the elongation, however, is greater for alloys containing less than 7% Zn or more than 15% Zn and less for alloys containing 7·5—14·5% Zn after the second annealing operation. The increase in the strength of zinc-aluminium alloys produced by quenching is less affected by subsequent heat treatment than that produced by deformation when the zinc content exceeds 5%, and more affected when the zinc is below 5%. To obtain the most satisfactory properties for commercial use the alloys should bo annealed above 275°; alloys containing 7·5—15% Zn especially should not be annealed below 275° as this decreases both tensile strength and ductility. A. R. Powell.

Aluminium and aluminium alloys. G. Sachs (Z. Metallk., 1926, 18, 209—212).—Ageing of duralumin or of aludur does not result in any change in the lattice structure as shown by Debye–Scherrer and Laue röntgenograms. *X*-Ray examination of twisted aluminium crystals indicates that the tendency of a metal to recrystallise is a function of the distortion suffered by the crystal lattice and has no relation to the hardness induced by heat treatment or cold work. Annealed aluminium alloys cannot be etched cleanly with a mixture of hydrochloric and hydrofluoric acids, whereas after heat treatment and ageing this acid mixture develops a bright etched surface. Tests on aludur indicate that the aged alloy is purer in a chemical sense than the annealed alloy, *i.e.*, that the precipitation of all or part of the constituents held in solid solution has not been effected by the ageing treatment, although such precipitation is usually held to account for the improvement in mechanical properties brought about by ageing. A. R. Powell.

Microstructure of duralumin alloys. A. Lennartz and W. Henninger (Z. Metallk., 1926, 18, 213—215).—The microstructure of alloys of the duralumin type can be satisfactorily developed by prolonged etching (6—24 hrs.) in concentrated nitric or sulphuric acid. An alloy containing 3·4% Cu, 0·47% Mg, 0·33% Si, and 0·25% Fe, after annealing at 500° and cooling slowly, showed when etched for 6 hrs. with cold nitric acid (*d* 1·4), an evenly distributed close network made up of small rounded particles in a white ground-mass of aluminium; after quenching from 500° and ageing for 6 days the structure consisted of large polyhedral grains with relatively few, small, rounded inclusions of a black constituent, and, after further annealing at 350°, precipitation of a disperse colloidal phase was indicated by the almost complete blackening of the specimen after 6 hrs. in nitric acid (*d* 1·4). Similar tests were made with an alloy containing 4·3% Cu, 0·65% Mg, 0·46% Si, 0·62% Fe, and 0·67% Mn; in this case the structure of the soft annealed alloy was not completely revealed by etching with nitric acid, and sulphuric acid etched the aged alloy differently to nitric acid. The former developed only a polyhedral structure, whereas the latter disclosed a coarse triplex structure which was not completely destroyed by subsequent annealing at 350°. A. R. Powell.

Aluminium alloys capable of being aged. W. Kroll (Metall u. Erz, 1926, 23, 225—230; Chem. Zentr., 1926, II., 640—641).—The effects of substituting other metals for magnesium, silicon, and copper in alloys of the duralumin type have been investigated. Copper may be replaced by 6%

of silver without affecting the ageing phenomena; substitution of lithium, rubidium, or cæsium for magnesium yields alloys which harden to a smaller extent than magnesium alloys on ageing. The other alkali metals, the alkaline-earth and the rare earth metals give alloys which do not exhibit ageing phenomena. Small quantities of the alkaline-earth metals are without influence on the tensile strength of lautal but they reduce the ductility slightly; the alkali metals, on the other hand, do not affect the ductility but slightly increase the tensile strength. Less than 0·5% of yttrium, lanthanum, or cerium improves the quality of lautal for rolling purposes, but otherwise has no effect; larger amounts have a deleterious influence on the metal. A. R. POWELL.

Influence of the compound $MgZn_2$ on the ageing of aluminium alloys. W. SANDER and K. L. MEISSNER (Z. anorg. Chem., 1926, **154**, 144—151).—Aluminium and the compound $MgZn_2$ form a quasi-binary system, the compound remaining undecomposed. The eutectic contains 25% Al and melts at 475°. At the eutectic temperature solid aluminium dissolves 28% of the compound, and the solubility falls to 4—5% at the ordinary temperature. The compound $MgZn_2$ thus behaves like Mg_2Si, and it may therefore be expected that alloys containing it will improve when kept. Alloys with 4—11% of the compound increase 50—70% in tensile strength when aged for 5 days at the ordinary temperature, the elongation diminishing slightly. Ageing at higher temperatures is much more rapid; an alloy containing 9% of the compound $MgZn_2$ after ageing for 10 hrs. at 80° has a tensile strength of 52 kg. per mm². and an elongation of 19%. A. GEAKE.

Conductivity changes during cold working [of tungsten] and their possible significance. W. GEISS and J. A. M. VAN LIEMPT (Z. Metallk., 1926, **18**, 216—218; cf. B., 1925, 361; A., 1925, ii, 372).—The electrical conductivity of single-crystal and polycrystalline tungsten wires changes by 3·5% for an elastic deformation of 1%, which is much less than the change caused by a corresponding amount of cold work, hence elastic strain cannot be the cause of the phenomenon. Again, the temperature coefficient of electrical resistance of cold-worked tungsten wires does not return to the normal after annealing at the recrystallisation temperature (1500°), although all elastic strains are then removed. As the ability of a metal to conduct electricity is connected in some way with the valency electrons it is suggested that the decrease of conductivity caused by cold work is due to deformation of the electron orbits. This explanation would account also for the increase in thermo-electric power and in electrochemical potential caused by cold work. Similar reasoning suggests that the allotropic change from α to β iron at 770° is due to a change in the configuration of the outer electrons. A. R. POWELL.

Plasticity of metals. I. H. SHOJI. **Plasticity of metals at high temperatures. II.** H. SHOJI and Y. MASHIYAMA (Sci. Papers Inst. Phys. Chem. Res., 1926, 4, 189—201, 202—205).—A new definition of the plasticity of metals is $P=(1/w_0).(dv/dw)$, where, when a metal is loaded by a constant weight so slightly beyond its elastic limit that the cross section of the metal is not altered by the tension, v is the velocity of the elongation per unit length, w_0 the intensity of the stress corresponding to the elastic limit where $v=0$, and w the intensity of the applied stress diminished by w_0. Determinations were made upon metals drawn into wires and annealed. The order of plasticity (diminishing) of metals at room-temperature is Cd, Pb, Sn, Au, Zn, Ag, Al, Cu. The plasticity of ductile metals, as compared with other metals, decreases more rapidly with the period of strain. In the cases of lead, tin, and cadmium the plasticity increases rapidly with rise of temperature, becoming very large as the melting point is reached. The elastic limit decreases with rise of temperature and becomes zero at the melting point. L. M. CLARK.

Addition of substances to pickling baths in the metal industries. W. H. CREUTZFELDT (Z. anorg. Chem., 1926, **154**, 213—219).—The loss of weight of iron and steel plates, and the increase in brittleness of wire, were determined after treatment under controlled conditions with sulphuric acid alone and with the addition of various organic substances known to moderate its action. The loss of weight was least when tar oil was added, and was 0·1—0·2 of the loss with sulphuric acid alone; many other organic substances retarded the action of the acid to a smaller extent. These substances are insoluble in the acid, and the retarding effect is only produced when they are finely dispersed and is therefore probably a physical or colloidal process. The pickling liquid is best prepared by first mixing the organic substance with concentrated sulphuric acid; the best temperature is 40°. A. GEAKE.

Determination of iron by means of titanium trichloride in the presence of copper. L. BRANDT (Stahl u. Eisen, 1926, **46**, 976—981).—Knecht and Hibbert's method for the determination of ferric salts by titration with titanous chloride using potassium thiocyanate as indicator fails if the solution contains copper owing to the reduction of the latter to cuprous salt and its subsequent precipitation as cuprous thiocyanate. Accurate results for iron may be obtained, however, by the use of the chromic acid compound of diphenylcarbohydrazide as indicator; titanium trichloride reduces this violet compound very slowly, but cuprous chloride reduces it instantly; hence a known volume of standard copper sulphate solution is added to the assay together with the indicator. For the determination of iron in ores, 1g. is dissolved in 50 c.c. of hydrochloric acid (d 1·19), with the addition, if ferrous compounds are present, of a small amount of pure manganese dioxide. The solution is diluted to 120 c.c., 5 c.c. of copper sulphate solution (1 c.c=0·001 g. Cu) and 0·7 c.c. of indicator are added, and the solution is titrated with standard titanium trichloride solution until colourless. If copper is also to be determined, a few c.c. of potassium thiocyanate solution are

added and titration continued until the red colour disappears. After making allowance for the copper added, the second amount of titanium solution corresponds with the copper in the ore. The indicator solution is made by dissolving 0·1 g. of *s*-diphenylcarbohydrazide in 15 c.c. of glacial acetic acid and diluting to 50 c.c. Immediately before use 4 c.c. of a 0·5% solution of potassium dichromate are added whereby an intense violet-red colour is produced ; the solution should not be kept more than a few hours. Platinum, antimony, and vanadium compounds do not interfere in the titration of iron by this method ; tungsten and molybdenum compounds, however, yield deeply coloured solutions which obscure the end-point. A. R. Powell.

Thermal and microscopical investigations of the cupola furnace slag systems $MnO-Al_2O_3-SiO_2$, $MnS-MnSiO_3$, and $CaS-CaSiO_3$. O. Glaser (Zentr. Min. Geol. Abt. A, 1926, 81—96 ; Chem. Zentr., 1926, II., 288).—The binary systems $Mn-SiO_2$, $Al_2O_3-SiO_2$, and $MnO-Al_2O_3$ were first examined and the results of earlier investigations confirmed. The system $MnO-SiO_2$ has a compound $3MnO,2SiO_2$ which decomposes at the melting point, as does the corresponding calcium compound. The addition of Al_2O_3 to mixtures of MnO and SiO_2 depresses the melting point up to a content of 20—25% Al_2O_3, a further addition causing it to rise again. The compound $2MnO,Al_2O_3,SiO_2$ melts at 1175°, while $MnO,Al_2O_3,2SiO_2$ melts at 1130°. The system $MnS-MnSiO_3$ shows in liquid form a limited solubility and in the solid form, as seen from metallographic investigation, complete immiscibility. The eutectic is at 6% MnS and melts at 1080°, while the gap in solubility is between 13 and 40% MnS. The system $CaS-CaSiO_3$ shows in the fluid condition a miscibility gap at 12—40% CaS, while in the solid state it seems to separate into its component parts. The eutectic is at 3% CaS and melts at 1440°. W. G. Carey.

Iron silicides. F. Körber (Z. Elektrochem., 1926, **32**, 371—378).—A review of the literature.

Colorimetric determination of molybdenum. Funck.—See A., Aug., 815.

Periodical heat treatment of gas cylinders. See I.

Patent.

Reduction of metal and making of [stainless iron and steel] alloys. W. Johnson (E.P 254,762, 6.2 and 18.7.25).—The slag on the metal in the usual open-hearth process of making steel is so adjusted that its lime content is 12—20% of the weight of the metal instead of the usual 6—7%, and its iron-oxide content 3—20% of the weight of chromite subsequently to be added. A preheated mixture of chromite and a non-carbonaceous reducing material, *e.g.*, ferrosilicon, is then charged into the furnace on to the surface of the slag, whereby an exothermic reaction takes place with the reduction of the greater part of the iron in the slag and of the chromium in the ore, with formation of an iron-chromium alloy of low carbon content. Other basic materials, *e.g.*, soda ash, may be added to the slag in order to prevent phosphorus being reduced back into the metal. A. R. Powell.

Purification of iron. G. S. Evans, Assr. to Mathieson Alkali Works (U.S.P. 1,590,739, 29.6.26. Appl., 10.4.25).—Molten iron is refined and desulphurised by maintaining an alkaline slag over its surface in a receptacle outside the melting furnace. Concurrently with the withdrawal of refined iron, fresh quantities of molten iron are transferred to the receptacle from the melting furnace, and fresh alkaline refining material is added to the slag as the operation proceeds. C. A. King.

Producing malleable iron. H. M. Williams and A. L. Boegehold, Assrs. to Gen. Motors Research Corp. (U.S.P. 1,591,598, 6.7.26. Appl., 21.5.24).—Molten iron containing a larger quantity of a constituent which precipitates carbon than is allowable in iron poured in sand moulds, and cooled at the usual rate, is poured into a mould and cooled rapidly to prevent the separation of flake graphite. The casting is then annealed. C. A. King.

Low-carbon steel for structural purposes. W. J. Crook, Assr. to Pacific Coast Steel Co. (U.S.P. 1,592,181, 13.7.26. Appl., 17.1.25).—A normal steel containing not more than 0·25% C and 0·5—1% Mn is heated at 700—900° for a period of 10 min. to 1 hr. or longer, quenched, reheated to 700° for a similar period, and then cooled either slowly or quickly. C. A. King.

[Iron-chromium] alloy. H. G. Weidenthal, Assr. to J. H. Herron Co. (U.S.P. 1,592,996, 20.7.26. Appl., 3.4.19).—A chromium-iron alloy containing 3—6% Cr, 1—2·5% Si, and less than 1% C is claimed. A. R. Powell.

Manufacture of iron-chromium alloys with low carbon content. Wargöns Aktiebolag (F.P. 602,298, 19.8.25. Conv., 18.9.24).—A mixture of chromite, silica, and carbon is smelted in an electric furnace to obtain an iron-chromium alloy with a high silicon content. This is added in the solid or, better, in the liquid state to a mixture of molten chromite and an aluminous material, such as clay, whereby the silicon in the alloy is replaced by more chromium and iron from the ore and a product containing less than 0·1% C and 2% Si is obtained. A. R. Powell.

Manufacture of iron and steel alloys. H. G. E. Cornelius (F.P. 603,049, 10.9.25. Conv., 24.9.24).—Briquettes are made of finely ground iron ore, sufficient carbon to reduce the iron oxide to metal and leave the desired content of carbon in the metal, and a suitable binder. Similar briquettes are made using the ore of the metal it is desired to alloy with the iron. The briquettes are smelted in an electric furnace either together or in any suitable order and the alloys formed are deoxidised with briquetted mixtures of manganese ore and carbon. A. R. Powell.

Production of titanium steel free from carbon. W. and H. MATHESIUS (G.P. 421,477, 25.3.21. Addn. to 408,668; cf. E.P. 221,529, B., 1924, 949).—For the reduction of the titania, magnesium or a metal of the alkali or alkaline-earth group, either alone or alloyed with iron, is used instead of the aluminium specified in the chief patent.

A. R. POWELL.

Manufacture of iron and steel. C. FLÖSSEL, and EISEN- & STAHLWERKE HOESCH A.-G. (G.P. 428,755, 27.5.24. Addn. to 381,006, B., 1924, 60).—The hot gases containing carbon monoxide derived from the converter treatment of pig-iron with air enriched with oxygen are used for the direct reduction of further quantities of iron ore in the same furnace in which the blowing of the iron is carried out so as to prevent heat losses during the passage of the gas to separate reducing chambers. A. R. POWELL.

Metallurgical furnace. A. HELFENSTEIN, and HELFENSTEIN-ELEKTRO-OFEN GES. (E.P. 231,203, 24.3.25).—In a metallurgical furnace having a relatively deep rectangular hearth, a mixture of fuel and the material under treatment is introduced along the two longer sides of the furnace so as to form a trough above the charge resting on the hearth. Pulverised or gaseous fuel is burned in the trough and considerable preheating of the charge takes place before it reaches the inner zone of the furnace. Combustible gases produced by reaction of the charge are burned in the cavity above, and at the same time the material sliding down the walls protects the latter from the destructive effects of heat.

C. A. KING.

Furnaces [for separating components of alloys], HÜTTENWERKE TEMPELHOF A. MEYER (E.P. 247,572, 10.10.25. Conv., 11.10.24).—In a cylindrical horizontal melting furnace for use in separating the components of alloys, *e.g.*, lead and tin alloys, a tapping tube extends so far through the side of the furnace as to penetrate a crust of cooling alloy and thus render possible the tapping of the fluid core. The furnace may be tilted about its longitudinal axis to complete the pouring of the core of molten alloy. C. A. KING.

Agglomeration of fine ores, roasted pyrites, coke smalls, and other pulverulent materials for smelting. BRÜCK, KRETSCHEL & Co., and O. KIPPE (E.P. 238,246, 7.8.25. Conv., 8.8.24).—From 3 to 5% of finely pulverised iron borings or other material containing metallic iron is mixed with the material to be agglomerated, and the mixture formed into briquettes, which are allowed to rust. A coherent and tenacious mass is obtained.

C. A. KING.

Apparatus for roasting, sintering and otherwise treating fuel-containing ore and the like by means of the Dwight and Lloyd process. METALLBANK & METALLURGISCHE GES. A.-G. (E.P. 244,733, 18.11.25. Conv., 17.12.24).—Two or more sintering or roasting pans are mounted on a rotary frame so that, on rotation, they register with charging, ignition, sintering, and discharging devices in sequence. The frame may be rotated intermittently to allow the material to be treated at any stage for any desired length of time. C. A. KING.

Electrolytic process for producing aluminium and its alloys. T. R. HAGLUND (E.P. 242,958, 5.10.25. Conv., 15.11.24).—The formation of hard crusts on the bath in the usual method of producing aluminium by electrolysis of molten fluorides containing alumina is avoided if the alumina is added as a mixture of 10—40% of the amorphous form and 90—60% of the crystalline form. The two varieties may be added simultaneously, or the crystalline form added first followed immediately by the amorphous form. A. R. POWELL.

Furnace for the production of aluminium and light metals by electrolysis of fused salts. VEREINIGTE ALUMINIUM-WERKE A.-G. (F.P. 603,726, 28.9.25. Conv., 7.10.24).—A furnace for the production of aluminium by electrolysis of fused salts comprises an iron vessel lined with graphite to serve as the cathode, and a cover carrying the anodes and provided with openings for removing the gases and for charging the furnace. The whole is mounted so that it can be rotated backwards and forwards or in one direction only about a vertical axis. A. R. POWELL.

Material resembling aluminium. E. SCHMID (G.P. 425,451, 21.3.24).—Fine aluminium powder or turnings are pressed into the desired shape under a pressure of at least 5000 kg./cm.^{2} and subsequently heated in an atmosphere of pure nitrogen to a temperature just below or well above the m.p. of aluminium. Alternatively the articles may be heated for some time well below the m.p. and subsequently heated further at about 1000°. A. R. POWELL.

Roasting sulphur-containing ores. G. ROSS (E.P. 248,704, 11.11.25. Conv., 9.3.25).—Sulphide ores are roasted in the usual way until most of the sulphur is removed; the residue is then separated electromagnetically or electrostatically into a final product poor in sulphur and a portion rich in sulphur. The latter is mixed with a fresh charge of ore for further roasting. In this way time is saved in the original roasting operation, as well as the fuel required to produce the higher temperature necessary for the final elimination of sulphur.

A. R. POWELL.

Manufacture of homogeneous, ductile pieces of chemically pure metals of the tungsten group having a high melting point, *e.g.*, tungsten, molybdenum, or uranium, and also of titanium and chromium. GEWERKSCHAFT WALLRAM ABT. METALLWERKE, H. VOIGTLÄNDER, and O. KAUFELS (E.P. 253,161, 7.2.25).—The brittleness of tungsten prepared by the ordinary methods is ascribed to the presence of small quantities of oxygen which fail to be removed by reduction in hydrogen. In order to remove this a small amount of a finely divided

reactive metal such as aluminium is mixed with the reduced metal and the mixture is heated to melt the tungsten and vaporise the aluminium oxide formed; alternatively the reduced metal may be heated in an atmosphere of aluminium vapour to such a temperature that the aluminium oxide formed volatilises. Another process for obtaining refractory metals from their oxides consists in reducing a mixture of the oxide with the oxide of another metal by the usual aluminothermic means, then separating the resulting alloy by known methods, *e.g.*, by centrifugal or magnetic forces while the metal is still in the molten state. Pure tungsten may also be obtained by causing two highly heated currents of hydrogen, one containing air-levigated particles of tungsten trioxide and the other atomised aluminium, to impinge on each other in a closed reaction vessel. A. R. Powell.

Manufacture of alloys. M. G. Korsunsky (E.P. 254,042, 14.4.25).—Alloys of copper containing 0·4—2·5% Co and 0·1—0·6% Si, or 1—10% Ni and 0·25—2·5% Si, with or without the addition of other metals, *e.g.*, tin and antimony, are amenable to heat treatment. Seven compositions are given as suitable for varying purposes. C. A. King.

Casting of magnesium and its alloys. G. Michel (E.P. 254,159, 17.10.25).—In the preparation of sand moulds for casting magnesium, the sand is kneaded with an oil in which resin is dissolved and to which also a solid hydrocarbon, *e.g.*, naphthalene, has been added. After the volatilisation of the oil by the molten casting, the resin forms a protective coating on the sand grains and the hydrocarbon acts as a preventive agent against the oxidation of the metal. C. A. King.

Alloys of platinum or palladium. M. G. Korsunsky (E.P. 254,666, 14.4.25).—Alloys of platinum or palladium, which are amenable to heat treatment, contain metals, such as chromium, iron, molybdenum, nickel, cobalt, tungsten, or tantalum, which form solid solutions with the precious metal, together with a smaller proportion of aluminium or silicon, so that part of the first metal added remains in the form of a silicide or an intermetallic aluminium compound in the alloy. If desired, a certain proportion of a third metal may be added to render the resulting alloy harder or more workable. The alloys are heat-treated by quenching them from 750—1000° and subsequently annealing at 300—500°. For example, an alloy containing 98·5% Pt, 1·2% Cr, and 0·3% Si has a Brinell hardness of 180 after heat-treatment, is readily workable, and has all the good properties of iridium-platinum alloys. A. R. Powell.

Gold alloys. M. G. Korsunsky (E.P. 254,979, 14.4.25).—Gold alloys amenable to heat treatment contain two elements which individually from solid solutions with gold and together form an intermetallic compound the solubility of which in gold varies with the temperature. One of these elements may be nickel, cobalt, chromium, iron, copper, palladium, or platinum, and the other is either silicon or aluminium. Further elements, such as copper or silver, may be added in order to obtain a definite carat. The final alloy must not contain more than 8% Ni, 6% Co, 6% Cr, or 4% Fe, nor less than 0·1% or more than 0·4% Si. After quenching from 750—1000° the alloys are soft (Brinell hardness about 75) and easily workable; by subsequent heat treatment at 200—600° they become hard (Brinell number 250) and tough. A. R. Powell.

Alloy for electrical contacts. Bell Telephone Manuf. Co. (Swiss P. 113,777, 14.8.24).—An alloy for electrical contacts comprises gold, silver, and such a proportion of at least one base metal that the alloy is sufficiently hard for the purpose required but has no tendency to develop a matt surface. A. R. Powell.

Flotation agent. A. L. Klees, Assr. to Combustion Utilities Corp. (U.S.P. 1,590,196, 29.6.26. Appl., 7.6.24). — Unsaturated hydrogenated and alkylated derivatives of aromatic hydrocarbons are used as flotation agents. C. A. King.

Concentration of mineral ores by flotation. F. T. Whitworth (U.S.P. 1,593,232, 20.7.26. Appl., 2.11.25).—In the concentration of ores by flotation a modifying agent consisting of the reaction product of an organic compound together with sulphur and phosphorus is added to the pulp. A. R. Powell.

Extracting nickel and cobalt from ferruginous ores. F. O. Kichline, Assr. to Bethlehem Steel Co. (U.S.P. 1,590,525, 29.6.26. Appl., 10.10.22).—A complex iron ore containing nickel or cobalt is mixed with an alkali sulphate and roasted at 680°. Nickel sulphate may be separated by lixiviation from the product, the sulphates of other metals being largely decomposed and rendered insoluble. C. A. King.

Production of oxides and other compounds of zinc, lead, and the like. L. E. Wemple, Assr. to Amer. Zinc, Lead & Smelting Co. (U.S.P. 1,591,253, 6.7.26. Appl., 17.2.20).—A mixed charge of metalliferous material and reduction fuel, is supported upon a bed of refractory material consisting of crushed gangue or clinker from a previous operation, saturated with volatilisable metal, and air is supplied to the charge to support combustion. Even penetration of the charge and emission of the metal as vapour are thereby attained. H. Royal-Dawson.

Ore reduction. Metallurgical process. J. E. Leonarz (U.S.P. 1,592,862-3, 20.7.26. Appl., [A] 17.9.21 and [B] 22.9.21).—(A) The ore is partially reduced by means of a gaseous reducing agent and the product is mixed with carbon and introduced into a reaction chamber over a bath of molten metal. The gases evolved are passed through the metal bath, then through highly heated coke, and utilised for the reduction of further quantities of ore. (B)

The ore is partially reduced with a gas, mixed with carbon, and added to a bath of the molten metal, the issuing gas being regenerated by passage through the molten metal. A. R. POWELL.

Recovering tin [from tinned iron]. E. G. HICKEY (U.S.P. 1,592,935, 20.7.26. Appl., 28.4.25). —In the recovery of tin from tinned iron scrap by the caustic soda process, ammonia is added as a catalyst. A. R. POWELL.

Volatilisation of metals from their ores. A. P. KNIGHT, Assr. to WESTERN METALLURGICAL Co. (U.S.P. 1,593,022, 20.7.26. Appl., 17.4.22).— The ore is heated in the presence of refractory material saturated with a substance which, under the action of the heat, yields a volatile compound with the metal in the ore. A. R. POWELL.

Crucible [for molten metal]. H. GEISENHÖNER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,593,399, 20.7.26. Appl., 18.5.23).—A crucible for molten metal is lined with bricks in such a way that alternate bricks project into the molten metal; by means of a tilting device the metal is caused to move over and between the bricks and is thus stirred thoroughly. A. R. POWELL.

Recovery of metal and other values from ores. A. E. SMAILL (U.S.P. 1,593,509, 20.7.26. Appl., 14.7.24. Conv., 3.1.24).—Sulphide ores are smelted with an alkaline flux under non-oxidising conditions to yield a slag and a self-disintegrating matte containing all the sulphur and metal values. A. R. POWELL.

Recovery of vanadium from ilmenite. RADIUM AND RARE EARTHS TREATMENT Co., Assees. of W. T. COOKE (Austral. P. 20,300, 30.10.24).— Ilmenite is heated with an equal weight of a mixture of sodium carbonate and sodium chloride for one hour at 800° which is sufficient to decompose the ore without melting the sodium carbonate. T. S. WHEELER.

Electrically insulating coating [on metal] affording protection against oxidation. E. WEINTRAUB, and SOC. ALSACIENNE DE CONSTRUCTIONS MÉCANIQUES (F.P. 600,774, 17.10.24).—Steel or other metals are heated in a powder of a simple or preferably complex tungstate, molybdate, antimonate, arsenate, or stannate, *e.g.*, a borotungstate or tungstomolybate, of iron, nickel, or copper when an adherent non-conducting coating which resists the action of oxygen is obtained. The coating can also be formed by immersing the metal in a bath of the molten coating agent, or by covering the metal with a paste of the agent and an agglomerating agent and heating, preferably in an atmosphere of an inert gas or under reduced pressure. These substances adhere also to glass so that they may be employed to make joints between metal and glass. T. S. WHEELER.

Production of tin free from lead from leady tin ores. Treatment of flue dust containing lead, zinc, and tin. METALLOCHEM. WERKE RODLEBEN A.-G. (F.P. [A] 603,474, 21.9.25, and [B] 603,408, 18.9.25).—(A) Tin ores containing lead compounds are leached with a hydrochloric acid solution saturated with sodium chloride which extracts the lead completely. (B) Flue dust, *e.g.*, that obtained by blowing scrap copper in a converter, is leached successively with hydrochloric acid, sodium chloride solution, and hot hydrochloric acid. The residual tin oxide contains only traces of lead and zinc. A. R. POWELL.

Separation by centrifugal force of alloyed or dissolved substances in the liquid state. H. JEGLINSKI (G.P. 428,552, 22.6.22. Addn. to 414,657; B., 1925, 927).—The zone in which the mother liquor or eutectic collects by reason of its density is heat-insulated, whilst the zone in which the primary crystals collect is allowed to cool normally. In this way a better separation is obtained. A. R. POWELL.

Treatment of ores or materials containing oxide of iron for recovering such oxide. S. G. S. DICKER. From KALMUS, COMSTOCK and WESCOTT (E.P. 255,639, 17.7.25).—See U.S.P. 1,552,786; B., 1925, 885. Bromine may be used in place of chlorine.

Producing ferro-chromium and other ferro-alloys. D. W. BERLIN, Assr. to AKTIEBOLAGET FERROLEGERINGAR (U.S.P. 1,593,157, 20.7.26. Appl., 20.7.23. Conv., 26.7.22).—See E.P. 201,520; B., 1924, 717.

Method of working refractory metals. A. LEDERER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,593,181, 20.7.26. Appl., 26.8.21. Conv., 29.12.15). —See E.P. 114,282; B., 1918, 272 A.

[Casting] of cast iron articles, parts of which have very different thicknesses. BRIT. PERLIT IRON Co., LTD. (E.P. 243,324, 20.10.25. Conv., 20.11.24).

Concentration of ores and the like [by flotation]. BAMAG-MEGUIN A.-G. (E.P. 238,241, 6.8.25. Conv., 6.8.24).

Production of carbon monoxide or water-gas (U.S.P. 1,592,860—1).—See II.

Recovery of tin from tinned iron (E.P. 253,958). —See XI.

XI.—ELECTROTECHNICS.

Water vapour pressure and electrical conductivity of wood. HASSELBLATT.—See IX.

Conductivity changes during cold working of tungsten. GEISS and VAN LIEMPT.—See X.

Synthetic manufacture of alcohol and acetic acid. PASCAL.—See XX.

PATENTS.

Electron-emitting electrodes. DUBILIER CONDENSER Co. (1925), LTD., Assees. of J. V. CAPICOTTO (E.P. 239,539, 5.9.25. Conv., 5.9.24).—

A heated platinum-iridium alloy is brought into contact with a mixture of barium and strontium carbonates, whereby the alloy becomes coated with barium and strontium oxides. J. S. G. Thomas.

Oxide cathodes for discharge tubes. Oxide cathode discharge tubes. N. V. Philips' Gloeilampenfabr. (E.P. [A] 245,145 and [B] 245,146, 23.12.25. Conv., 24.12.24).—(A) A base metal core oxidised superficially, wholly or partly, is coated with an alkaline-earth azido or a mixture of such azides, and is then heated to such a temperature that the azide decomposes. The core is finally heated in an atmosphere containing oxygen until the layer of alkaline-earth metal is at least partly oxidised. (B) In a discharge tube are arranged a metal body wholly or partly superficially oxidised, and adapted to form a support for the active oxide layer of the cathode, together with one or more electrodes on which there is a compound or a mixture of compounds from which one or more alkaline-earth metals are liberated on heating. These electrodes are heated by currents induced therein by a high-frequency magnetic field so that the alkaline-earth metal or metals volatilise and are deposited on the metal body, and the deposit is oxidised, preferably while the discharge tube is being exhausted. J. S. G. Thomas.

Lead-in wires for electric lamps and the like. Metal and Thermit Corp., Assces. of K. W. Schwartz (E.P. 249,084, 1.2.16. Conv., 10.3.25).—Nickel steel wires of the desired size and having a suitable coefficient of expansion are cleaned by pickling in alkali, acid, and alkali baths in succession, and plated with chromium from a bath containing chromic acid and a chromium salt in the ratio of at least 2 : 1. The coated wire is then annealed at about 320° in an oil bath to remove occluded hydrogen. A. R. Powell.

Coating the internal metal parts of electric lamps with chemical preparations. Elektrische Glühlampfabr. "Watt" A.-G. (Austr. P. 102,539, 4.11.24).—The wires, after sealing into the lamps, are dipped into a suspension of finely divided phosphorus in water which has been previously given an electrical charge, and a current is passed through the wires so as to set up a potential difference between them and the suspension. In this way the thickness of the coating produced may be exactly regulated. A. R. Powell.

[Recovery of tin by] electrolysis [from tinned iron]. M. A. Adam (E.P. 253,958, 1.1.25).—For the recovery of tin from tinned iron scrap by the process described in E.P. 154,242 (B., 1921, 87 A) the apparatus described in E.P. 154,635 (B., 1921, 49 A) is modified in that the means for removing the loose deposit of tin from the cathode consist of rubber belts or the like provided with ebonite or vulcanite blades at intervals and moving at right angles to the travel of the periphery of the cathode. The anode consists of graphite blocks rendered waterproof by impregnation with paraffin wax facing similar blocks of unimpregnated graphite. A. R. Powell.

Electrolytic decomposition of alkali chloride solutions. Königsberger Zellstoff-Fabr. & Chem. Werke Koholyt A.-G., and E. Schlumberger (E.P. 254,163, 24.10.25).—In order to reduce the rate of corrosion of the graphite anodes used in the electrolysis of solutions of alkali chlorides the anodes are made very porous and are provided with internal channels or borings through which the salt solution is passed, so that it can flow through the pores of the electrodes into the bath. A. R. Powell.

Electrode for electrolytic decomposition apparatus. C. F. Holmboe (U.S.P. 1,588,579, 15.6.26. Appl., 1.10.25. Conv., 4.7.25).—The ribs of the electrodes described in E.P. 221,189 (B., 1925, 511) are provided with fin-like projections to lessen the distance between the electrodes and diminish the electrolytic resistance. E. S. Kreis.

Process for bringing about physical and chemical changes in liquid or pasty dielectric carbon compounds by means of ionised gases. Oelwerke Stern-Sonneborn A.-G., and H. Vogel (E.P. 254,375, 30.3.25).—Liquid or pasty dielectric carbon compounds are mixed with gases and subjected to large alternating currents of high voltage or high frequency and of high ionising power, in an electric condenser having stationary electrodes provided with an additional dielectric, whereby molecular changes are produced in the carbon compounds, and minute subdivision of the material resulting in the formation of froth is effected. Applications of the process mentioned are the hydrogenation of fats, the oxidation of alcohols, aldehydes, etc., the production of lubricating oils from mineral oils by polymerisation. J. S. G. Thomas.

High frequency induction furnaces. C. R. Burch, N. R. Davis, and Metropolitan-Vickers Electrical Co., Ltd. (E.P. 255,157, 17.4.25).

Electroplating baths for simultaneously obtaining metallic deposits of various thicknesses. Württembergische Metallwarenfabr., and A. Wolf (E.P. 255,691, 19.10.25); Württembergische Metallwarenfabr., A. Wolf, and E. Bauer (E.P. 255,736, 25.1.26).

See also pages 736, **Aromatic aminohydroxy-compounds** (E.P. 254,204). 738, **Films from cellulose solutions** (E.P. 254,946). 744, **Nitrides** (Austr. P. 103,216). 746, **Electrically insulating glass** (E.P. 254,174–5). 754, **Aluminium and its alloys** (E.P. 242,958); **Production of aluminium etc.** (F.P. 603,726). 755, **Alloy for electrical contacts** (Swiss P. 113,777). 756, **Insulating coating on metals** (F.P. 600,774). 759, **Purification of soap solutions** (G.P. 424,679).

XII.—FATS; OILS; WAXES.

Free acids of edible oils [olive oil]. A. Ceriotti and A. Sanguinetti (Rev. Fac. Cien. Quím., 1925, 3, 65—75).—Data are given for the

free acid content, expressed as oleic acid, of samples of olive oil from different countries. Since free acidity greater than 1·5% is only shown in a few cases, it is recommended that this figure should be adopted as the legal maximum. G. W. ROBINSON.

Composition of herring oil. I. Saturated acids and acids of the oleic series of "ô-nishin" (great herring) oil. M. TSUJIMOTO (J. Soc. Chem. Ind. Japan, 1926, 29, 195—202).—The oil used for the experiments had the following characteristics: d_4^{15} 0·9167, n^{15} 1·4731, acid value 6·53, saponif. value 185, iodine value 100, Hehner value 95·4, Reichert-Meissl value 0·73, unsaponifiable matter 1·01%. From the saturated acids (about 19% of the total acids), palmitic and myristic acids were isolated, the former being the main constituent. Stearic and higher saturated acids were present in far smaller amount. The unsaturated acids consist mainly of acids of the oleic series (probably up to 80%). $C_{16}H_{30}O_2$ (neutralisation value 214·5, iodine value 94·5, zoomaric acid?), $C_{18}H_{34}O_2$ (neutralisation value 199·8, iodine value 90·3, probably ordinary oleic acid), $C_{20}H_{38}O_2$ (neutralisation value 176·1, iodine value 82·3, gadoleic acid?), and $C_{22}H_{42}O_2$ (m.p. 33°, neutralisation value 165·2, iodine value 75·2, probably identical with Toyama's cetoleic acid) were isolated. Hydrogenation experiments indicated that the acids of the oleic series were straight-chain compounds. About 30—40% of the total fatty acids are C_{20} and C_{22} acids of the oleic series. K. KASHIMA.

Determination of the composition of wood [tung] oils with help of the thiocyanogen value. H.P. KAUFMANN (Ber., 1926, 59, 1390—1397).—Addition of thiocyanogen to compounds containing ethylenic linkings is even more markedly selective than that of bromine. In glacial acetic acid-carbon tetrachloride in the dark, addition takes place with ethylene, butylene, phenylacetylene, tolane, anethole, safrole, allyl alcohol, oleic, elaidic, erucic, brassidic, ricinoleic and ricinelaidic acids, ethyl acetoacetate, acetyldibenzoylmethane, ethyl diacetylsuccinate (α, β form), ethyl formylphenylacetate (γ form), antipyrine, and pinene, but not with acetylene, styrene, stilbene, crotonic, fumaric, maleic, and cinnamic acids (or their esters), cinnamaldehyde, ethyl diacetylsuccinate (α_2 β and α forms), stearolic and behenolic acids. Since linoleic acid, its methyl ester and glyceride add 2 mols. of bromine but only 1 mol. of thiocyanogen, the proportion of linolein in glycerides of fatty acids which add thiocyanogen quantitatively (*e.g.* oleic acid), and indifferent substances in an oil can be deduced from the thiocyanogen and bromine values. Elæostearic acid, which polymerises much more readily than has been suspected previously, absorbs only 1 mol. of thiocyanogen whereas 2 mols. of bromine are added from solution in methyl alcohol saturated with sodium bromide and 3 mols. from solutions in carbon tetrachloride exposed to light (thus confirming the constitution $CH_3{\cdot}[CH_2]_3{\cdot}[CH{:}CH]_3{\cdot}[CH_2]_7{\cdot}CO_2H$ for the acid). The composition of various samples of tung oil, deduced from the iodine and thiocyanogen values, is elæostearin 78·5—87·1%, olein 8·5—22·8%, and, in some cases, glycerides of saturated acids and unsaponifiable matter. H. WREN.

Marine animal oils. Calamary [*Todarus sagittatus*, Lk.] oil. E. ANDRÉ and H. CANAL (Compt. rend., 1926, 183, 152—154; cf. B., 1926, 247).—The hypothesis that sperm whale oil derives its peculiarities from the fats in the cephalopods eaten by the whale is not supported by a study of the fats of the calamary (squid), which is nearly related to the large molluscs actually eaten by the sperm whale. The fat extracted from all parts of the calamary has d 0·93—1·01, iodine value (Hanus) 144·5—173·6, saponif. value 135·7—163·4, fatty acids 75—88%, unsaponifiable matter 8·0—25·0%, acetyl value 28·7—94·2. The unsaponifiable portion is cholesterol and contains no hydrocarbons. The acids have saponif. value 187·5—190·3, iodine value (Hanus) 175·4—205·2, mean mol. wt. 294—298, and contain members of the clupanodonic acid series. Calamary fat does not contain aliphatic saturated or unsaturated alcohols, but consists of glycerides and a high proportion of cholesterides. G. M. BENNETT.

Seeds of *Euphorbia helioscopia*, L. P. GILLOT (Bull. Sci. Pharmacol., 1926, 33, 193—196; Chem. Zentr., 1926, II., 44).—The seeds contain water, 7·74; fat, 32·61; protein 17·43; sugar, 2·18; mineral matter, 6·22; and cellulose, 33·82%. The oil yielded on pressing or extraction with light petroleum has d_{15}^{15} 0·9346, n_D^{15} 1·4847, acid value 0·6 (=0·33% of oleic acid), soluble fatty acids equivalent to 0·06% of butyric acid, insoluble fatty acids and unsaponifiable matter 95·67%, saponification value 191·1, iodine value (Wijs) 204·4; unsaponifiable matter 0·7%, ether-insoluble bromides (Hehner and Mitchell) 58·4%. The total fatty acids have n_D^{40} 1·4670, iodine value 213·5. The oil resembles the oils from *Mercurialis* varieties, is strongly purgative, and has drying properties. B. FULLMAN.

Bleaching action of 60% and 35% hydrogen peroxide on saponifiable oils and fats. C. STIEPEL (Seifensieder-Ztg., 1925, 53, 291—292; Chem. Zentr., 1926, II., 125).—The bleaching effect of hydrogen peroxide depends solely on the actual amount of peroxide present and not on the concentration; the reaction can be carried out at a lower temperature if 30% peroxide is used. Intimate mixing is essential for successful operation. B. W. CLARKE.

Colloid reactions in the oil and fat industries. J. LEIMDÖRFER (Seifensieder-Ztg., 1925, 52, 791—792, 835—836, 854—855, 874—875, 893—894, 912—913, 931—932, 950—951, 968—969, 987—989; Chem. Zentr., 1926, II., 124).—Colloidal characteristics are of greater importance in connexion with the digestibility of fatty foodstuffs, *e.g.*, milk, butter, and margarine, than chemical homogeneity. The extraction and refining processes for oils, such as olive oil, are determined by the colloidal nature of the oil. When the oil is not to be subjected to further refining

operations the removal of free fatty acids by means of alcohol is recommended. The hardening of fats by catalytic hydrogenation depends upon reactions in the colloidal state. The "poisoning" of catalysts is due to a change in colloidal conditions, and the worst "poisons" are too high temperatures and pressures. B. W. CLARKE.

Polymerisation of fatty oils. H. PFAHLER (Chem. Umschau, 1926, 33, 173—176).—Thickened soya bean and linseed oils were saponified, and the fatty acids were isolated and then distilled *in vacuo*. From the former oil there were thus obtained in the distillate 32·5% of the total acids, consisting of 11·7% of saturated acids, 17·6% of oleic acid, and 3·2% of a dibasic unsaturated acid. The original oil contained 9·35% of saturated acids, 30·8% of oleic acid, and 55·4% of linoleic and linolenic acids. It is suggested that a dibasic unsaturated acid and a neutral unsaturated compound may arise from the splitting of a dimeride across the two original double linkings that form a 4-carbon ring in the polymer. The mol. wt. of the residual fatty acids (64% of the total fatty acids) is too low for complete polymerisation to have occurred. Linseed oil, thickened almost to the gelatinising stage, and treated by this method, gave a distillate consisting of 8% of saturated acids, 22·5% of oleic acid (2·5% of *iso*oleic acid), 12% of linoleic acid, and approx. 0·75% of a dibasic unsaturated acid. The distillation residue (53% of the total fatty acids) had mol. wt. 408, which indicates the partially polymerised state of the linolenic and linoleic acids of which it was composed. The possible polymerisation-inhibiting effect of the relatively stable oleic acid in such mixed glycerides as dioleolinolein, linoleodiolein, etc. is discussed.
S. S. WOOLF.

Measurement of the emulsifying power of soap solutions by means of the drop-number. D. M. SIMM (J. Soc. Dyers and Col., 1926, 42, 212).—Comparison of the emulsifying powers of various textile soaps by means of Donnan's drop-number apparatus was not satisfactory since results obtained with 2-c.c. and 5-c.c. pipettes rarely agreed and the drop-numbers increased with the age of the soap solutions as found by Cobb (B., 1926, 998). The minimum time of ageing necessary to produce an alteration of drop-number varied from 10 to 150 min. with different soaps. A. J. HALL.

See also A., Aug., 816, **Unsaponifiable matter from oils of elasmobranch fish. Constitution of squalene (spinacene)** (HEILBRON, KAMM, and OWENS). 819, **Fatty acid derivatives** (WHITBY). 866, **Ester-hydrolysing actions of the castor bean** (LORBERBLATT and FALK).

Preparation of fuel oil by distillation of soaps of soya bean oil (SATO and TSENG).—See II.

Colour of oils etc. FONROBERT and PALLAUF.—See XIII.

PATENTS.

Treatment of crude cholesterol [wool fat] materials and manufacture of anti-corrosive preparations. S. FOWLER and E. EDSER (E.P. 253,995, 24.3.25).—In the purification of wool fat (*adeps lanæ*) by converting the fatty acids present in the crude material into soluble soaps by treatment with alkali and subsequent washing, a large proportion of the wool fat becomes emulsified in the soap solution and cannot be completely recovered. If, however, the purification be effected in successive stages, using quantities of sodium peroxide sufficient only for incomplete saponification, and removing the soaps formed at each stage, a wool fat substantially neutral and free from soaps results, suitable for anti-rust coatings. A fully detailed example of the process is given. S. S. WOOLF.

Purification of soap solutions. H. HARRIES, Assee. of C. HARRIES (G.P. 424,679, 21.7.22).—Soap solutions are freed from unsaponified impurities by dialysis (or electro-osmosis) through a suitable membrane. A. J. HALL.

Separation of oils from soap solutions. H. HARRIES, Assee. of C. HARRIES (G.P. 425,629, 29.10.22. Addn. to 424,409; B., 1926, 594).—The treatment described in the chief patent is carried out more rapidly, cheaply, and completely if a series of cells is used instead of one cell. A. J. HALL.

Bringing about changes in dielectric carbon compounds (E.P. 254,375).—See XI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Testing of paint pigments for transparency to ultra-violet radiation. G. F. A. STUTZ (J. Franklin Inst., 1926, 202, 89—98).—The absorption of ultra-violet light by paint films has been shown to cause rapid deterioration (cf. B., 1925, 770). In the present paper the ratio of transmitted to reflected light for a large number of pigments and inerts is given for the range 3023—4358 A. A layer of the pigment 0·00092 mm. thick in castor oil, which has practically no absorption in this range, was used, and the transmitted and reflected light were caught in two integrating spheres, and measured with a quartz spectrograph. Linseed oil absorbs completely rays of wave length less than 3700 A, and becomes brittle and crumbles. A pigment should therefore absorb or reflect all ultraviolet light. The thickness of layer necessary for various pigments, and whether their opacity is due to absorption or reflection, is shown in tables and curves. In order to be completely opaque to light rays shorter than 3700 A., zinc oxide must be used in a layer 0·00092 mm. thick, carbon black 0·00062 mm., lithopone 0·01 mm., and white lead 0·027 mm. The average three-coat paint film is about 0·12 mm. thick and contains sufficient pigment to form a layer 0·035—0·045 mm. in thickness. Aluminium dust and zinc dust in a layer one particle thick (0·004—0·007 mm.) are entirely opaque to light of all wave-lengths.
C. J. SMITHELLS.

Examination of inorganic pigments in the dark field of the analytical quartz-lamp. A.

EIBNER, L. WIDENMAYER, and A. STOIS (Farben-Ztg. 1926, 31, 2399—2400).—When pigments are subjected to pure (invisible) ultra-violet rays (by the use of a dark filter in conjunction with a quartz lamp) fluorescence, phosphorescence, or neither may be caused. The behaviour under this treatment of a large number of white and coloured, mineral and manufactured pigments, both transparent and opaque, is recorded and the possibilities of the method are discussed. S. S. WOOLF.

Scope of the analytical quartz-lamp in the paint and varnish industry. K. SCHMIDINGER (Farben-Ztg., 1926, 31, 2451—2452; cf. preceding abstract).—The fluorescence emitted by substances under the action of invisible ultraviolet rays can be utilised for the estimation of quality, detection of adulteration, etc. The natural colours and characteristic fluorescences induced under the ultraviolet (dark filter) lamp, of many solvents, resins, pigments, and various other products used in the paint and varnish industry are tabulated. S. S. WOOLF.

Optical examination of pigments. A. V. BLOM (Farbe u. Lack, 1926, 247—248).—The microscopical examination of pigments by polarised light permits the detection of crystalline, cryptocrystalline, and truly amorphous particles, and can be used successfully to elucidate problems of freshly-applied and aged paint-films. Six photomicrographs are reproduced and discussed. Red oxide of iron and "Subox," the recently described rust-preventive lead suboxide pigment, are shown to be heterodisperse mixtures of isotropic and anisotropic components. The behaviour of an ordinary and a highly-dispersed red lead, when mixed with linseed oil of rather high acid value, indicates that the non-settling properties induced by high dispersion are counteracted by a tendency to produce hard deposits of lead soaps owing to the increased activity of the particles.
S. S. WOOLF.

"Leading" by [lead suboxide] paint. A. V. BLOM (Farben-Ztg., 1926, 31, 2401—2402).—Non-settling qualities and low initial content of oxygen lengthen the life of lead suboxide paint films. Suspensions in oil of ordinary and specially dispersed red leads were compared with priming and covering coats of "leading paints," After 24 hrs., when the ordinary red lead had completely settled and the dispersed red lead pigment had settled to over ⅓ of the total column, practically no sedimentation was shown in the suspensions containing lead suboxide. The specific gravities of the pigments are approximately the same, and the dispersed red lead has smaller sized particles than the lead suboxide, the fine structure of the latter conferring its especial non-settling properties. Imponderable weights of lead (a unimolecular film of lead) suffice to protect iron surfaces from rust, so that in practice it is not necessary to use the most active modifications of the pigment (liable to pyrophoric oxidation). More stable forms are obtainable. The author's experiments on the non-rusting of "leaded" plates when scratched with a penknife and exposed to the action of water and air, indicate a passivity of the iron in which iron-lead elements play a part. S. S. WOOLF.

White lead pigments. K. WÜRTH (Farbe u. Lack, 1926, 134—135, 170, 183).—A résumé of the properties of basic carbonate and basic sulphate of lead, with stress on the lead poisoning aspect. The U.S. specifications for these pigments are quoted in extenso. S. S. WOOLF.

New method of determining the colour of resins, varnishes, oils, etc. E. FONROBERT and F. PALLAUF (Farbe u. Lack, 1926, 231—232).—A concise summary of the applications and method of determination of "iodine colour values," previously described (cf. B., 1926, 201, 450). S. S. WOOLF.

Polymerisation of fatty oils. PFAHLER.—See XII.

PATENTS.

Zirconium oxide compound [pigment]. C. J. KINZIE, Assr. to TITANIUM ALLOY MANUF. Co. (U.S.P. 1,588,476, 15.6.26. Appl., 5.5.25).—A compound containing zirconium is heated to obtain all the zirconium as oxide and then mixed with less than 15% of sodium silicofluoride and heated to 1000° for 3 hrs. to decompose impurities. The resulting zirconia is white and suitable for use as a pigment. E. S. KREIS.

Nitrocellulose varnishes. GROSS, SHERWOOD & HEALD, LTD., R. A. PHILLIPS, and H. A. R. THURLOW (E.P. 254,041, 9.4.25).—One part of nitrocellulose having a nitrogen content of approx. 12%, is added to 1—3 pts. of a cyclic ketone (*e.g. cyclo*hexanone) or an ester of a cyclic alcohol (*e.g. cyclo*hexanyl formate or acetate), and the whole heated for ½ to 1½ hrs. at 40° to form a homogeneous viscous solution. This is thinned with benzene, white [petroleum] spirit, etc., and resins (*e.g.* elemi, thus, copal), castor oil, and plasticisers (*e.g.* camphor, butyl tartrate) are added. A varnish or, if pigment be incorporated, an enamel, is thus obtained that can be applied with a brush. S. S. WOOLF.

Treating [drying] oil. H. P. TABER (U.S.P. 1,590,656, 29.6.26. Appl., 19.8.21).—By utilising convection currents, a drying oil is alternately heated and exposed to atmospheric air, in a tank provided with vertical baffle-plates. S. S. WOOLF.

Preparation of pale-coloured condensation products of phenols and formaldehyde [artificial resins] stable to light and air. O. EHRLICH (G.P. 423,032, 10.5.22).—The spongy intermediate products (resols) obtained in the treatment of phenols with formaldehyde are treated with substances, such as lactic, acetic, tartaric, or boric acid, which cause them to deflate and allow of the ready removal of the excess unchanged phenol which is retained in the resin-phenol emulsions usually obtained. For example the resinous condensation product obtained by heating phenol with 40% formaldehyde and sodium carbonate is washed with hot water until it becomes thick and viscous, then shaken vigorously three or four times for 5 min. with 4% acetic acid at 25—30°. The

resulting thin resin is washed again with water and, after addition of a small quantity of ammonia, is dewatered in a vacuum and hardened in the usual way. The resulting transparent, colourless or faintly yellow resin is insoluble in acids and alkalis and, owing to the absence of phenol, stable to light and air. A. R. POWELL.

Rust preventive coatings. R. EBERHARD (G.P, 425,900, 13.11.24).—Nascent chromium chromate is combined with various oils, waxes, resins, varnishess, solvents, etc. Chromyl compounds and molybdic acid, preferably in combination with tungstic anhydride and soluble chlorides, in similar media, are also claimed. S. S. WOOLF.

[Paint and varnish] bases. BERGOLIN-WERKE W. VAN DEN BERGH (G.P. 427,411, 15.3.22).—Rosin is incompletely neutralised by successive heating with 4% of lime to 132°, and with about 4% of glycerin to 193°. The mass is then thinned with solvents to a consistency suitable for brushing and fillers are added. S. S. WOOLF.

Manufacture of [resinous] phenolic condensation products. H. WADE. From S. KARPEN & BROS. (E.P. 255,692, 23.10.25).—See U.S.P. 1,566,823; B., 1926, 202.

Obtaining pure gum [resin] from some crude gum materials. N. BUCHAN (U.S.P. 1,593,964, 27.7.26. Appl., 18.6.25. Conv., 23.7.24). —See E.P. 237,576; B., 1925, 965.

Bituminous emulsions (E.P. 254,004).—See IX.

Anti-corrosive preparations (E.P. 253,995).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Elastic properties of raw rubber in relation to packing density. M. KRÖGER (Gummi-Ztg., 1926, 40, 2373—2376).—The load-extension curves of samples of rubber of various histories have been plotted for various temperatures; the results are discussed. S. I. LEVY.

Natural ageing test [of vulcanised rubber]. H. P. STEVENS (Bull. Rubber Growers Res. Assoc., 1926, 8, 345—346).—Elongation and breaking-load tests carried out in June, 1926, with vulcanised samples prepared in March, 1922, from rubbers coagulated with acetic acid and with varying proportions of sodium silicofluoride show that the latter age better; coagulation with silicofluoride gives slower curing rubbers than coagulation with acetic acid. The tests confirm results obtained with artifically aged rubbers. S. I. LEVY.

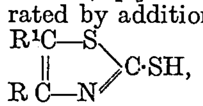
Experiment to show that the sub-permanent set of vulcanised rubber decreases with increased period of vulcanisation. T. YAMASAKI (J. Soc. Chem. Ind. Japan, 1926, 29, 127—132). —From results obtained with four samples composed of different amounts of smoked sheet rubber, zinc oxide, sulphur, hexamethylenetetramine, calcium carbonate, talc, and magnesium carbonate, and also with two rubber insulation stocks, it is concluded that the subpermanent set of vulcanised rubber, contrary to the results of Kratz and Flower (cf. B., 1919, 151 A), decreases with increased period of vulcanisation even in the presence of mineral fillers; this is true not only for specially compounded rubber, but also for insulating rubber stocks. The absolute value of the sub-permanent set varies considerably with the kind of mineral fillers used. The contraction after release of the tension is quite regular. K. KASHIMA.

Structure of stretched rubber. I. E. A. HAUSER and H. MARK (Koll.-Chem. Beih., 1926, 22, 63—94).—A detailed account of an investigation, the chief results of which have already been published (cf. B., 1926, 334).

See also A., Aug., 840, **Formation of *cyclo*-caoutchouc from caoutchouc hydrogen halides** (STAUDINGER and WIDMER). 841, **Behaviour of caoutchouc on heating** (STAUDINGER and GREIGER); **Resin of *Hevea* rubber** (WHITBY, DOLID, and YORSTON).

PATENTS.

Vulcanisation of caoutchouc. L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. [A] 1,581,439 and [B] 1,491,441, 6.7.26. Appl., [A] 5.11.23, [B] 16.3.26).—(A) Vulcanisation is accelerated by addition of a substance having the formula



or any of its derivatives.
(B) The substance used for acceleration has the structure given under (A), but R and R^1 are similar radicals. S. I. LEVY.

Vulcanised material. G. A. HENDERSON (U.S.P. 1,591,454, 6.7.26. Appl., 10.1.22. Renewed 8.5.26). —Steam carrying sulphur in suspension (preferably formed by interaction of hydrogen sulphide and sulphur dioxide) is injected into the heated material to be vulcanised, and the mass is agitated so as to form thin films. S. I. LEVY.

Distillation of waste rubber. P. BAIRGOIS (F.P. 603,772, 19.12.24).—Vulcanised rubber containing 30—50% of mineral fillers is mixed, before distillation, with a stable substance which is fluid but not volatile at 350—360°, *e.g.*, pitch or paraffin wax, so that the residue in the distillation retort is fluid and can be run out. A. J. HALL.

Compounding of rubber and the like. MORGAN & WRIGHT, Assees. of E. E. A. G. MEYER (E.P. 249,065, 4.8.25. Conv., 13.3.25).—See U.S.P. 1,558,701; B., 1926, 22.

Bituminous emulsions (E.P. 254,004).—See IX.

XV.—LEATHER; GLUE.

PATENTS.

Tanning animal hides. H. STINNES-RIEBECK-MONTAN & OELWERKE A.-G. (G.P. 423,096, 12.3.22).

—The raw hides are treated with the products obtained by esterifying sulphite-cellulose waste lye with aromatic sulphonyl chlorides, if necessary mixed with other synthetic or natural tanning agents. The ester obtained by mixing sulphite-cellulose lye (d 1·26) with sodium hydroxide and p-toluenesulphonyl chloride at 15° and heating to 100° is an excellent precipitant for glue, and gives improved tanning results when mixed with known synthetic tanning agents, giving better plumping.

R. BRIGHTMAN.

Preparation of chromium oxide compounds [for tanning]. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of K. DAIMLER (G.P.423,138, 2.4.22).—Chromium trioxide or its salts are reduced with lignic acid, the latter being obtained by alkaline extraction of wood or straw, waste lye from the soda-cellulose process being especially suitable. Soluble degradation products of lignic acid are also formed, which render the chromium sesquioxide solution less sensitive towards neutralisation with alkali, and hence more valuable to the tanner than a pure chrome alum solution. For instance, waste lye from the manufacture of straw cellulose (40% of dry solids) is diluted with water and precipitated with 50% sulphuric acid at 20°. A warm mixture of sulphuric acid and potassium dichromate is run in during 3—4 hrs. at 80—90°. After filtering and washing the brown residue with hot water, the filtrate and washings are united and the bluish-green solution is evaporated to dryness.

R. BRIGHTMAN.

Purification of industrial effluents (F.P. 597,515).—See XXIII.

XVI.—AGRICULTURE.

Theory of the origin of alkali soils. A. A. J. DE'SIGMOND (Soil Sci., 1926, **21**, 455—480).—The formation of alkali soils in general is discussed from the standpoint of climatic and geological conditions. The question is raised as to whether a superabundance of sodium salts is an essential factor in the formation of such soils.

A. G. POLLARD.

Moisture equivalent of soils. M. D. THOMAS and K. HARRIS (Soil Sci., 1926, **21**, 411—424).—The Briggs–McLane method for determining the moisture equivalent of soils (U.S. Bur. Soils Bull. 45, 1907) is shown to suffer certain limitations both in the actual mechanical process and in the theoretical aspects. The size of the sample influences the amount of water retained after centrifuging. Very fine and very coarse soils retain slightly less water when larger samples are used. This effect is more marked in soils of an intermediate texture. Similarly the moisture gradient in the soil mass opposing the centrifugal force is greatest in soils of medium texture. Clays and very fine silts reach capillary equilibrium only after centrifuging for many hours. Silts of 10μ mean diameter retain more water than samples of 5μ mean diameter and the difference decreases with increasing bulk of sample taken. Very fine silt retains more water than clay. It is suggested that the capillaries in the former are completely full of water, but those in the latter contain a proportion of colloidal matter. The drying of the soil mass is affected by, and follows theoretically, the reduction in the size of the capillaries in the boundary surfaces. The impermeability, and probably the moisture-retaining power of clay is greater when the replaceable base is entirely sodium, than when potassium, ammonium, calcium, aluminium, or hydrogen is present. The colloidal swelling of the complex is increased by the presence of sodium.

A. G. POLLARD.

Do colloids exist as a coating round the soil grains? G. J. BOUYOUCOS (Soil Sci., 1926, **21**, 481—487).—Studies of the colloid content of soils are based upon determination of the heats of wetting. It is concluded that the colloids exist not only as coatings of mineral particles of soils, but also as a free and separate component scattered irregularly through the soil mass. Soil material passing 200- and 325-mesh sieves is not clay, but frequently contains less clay and colloids than the coarser mass remaining.

A. G. POLLARD.

Method for study of the reaction of soils in regard to alkalis. J. CLARENS (Bull. Soc. chim., 1926, [iv.], **39**, 795—802).—A series of 2 g. samples of the soil are treated with increasing amounts of 0·1N-sodium hydroxide (0—4 c.c.) and left, with gentle shaking, until equilibrium is established. Each mixture is then shaken with a large excess of quinol, in the presence of oxygen also in excess; and the oxygen-absorption is measured after 3½ hrs. The apparatus used has been previously described (cf. B., 1921, 785 A). Under these conditions, the oxygen absorbed by oxidation of the quinol can be used as a measure of the amount of alkali present (cf. A., 1924, i, 1379). By plotting the volumes of oxygen absorbed against the amounts of alkali originally added to the soil, a curve is obtained which is compared with the curve given in absence of soil. The effect of various soil treatments on the form of the curve may be studied.

C. T. GIMINGHAM.

Relation of fineness of grinding to rate of sulphur oxidation in soils. R. E. STEPHENSON (Soil Sci., 1926, **21**, 489—494).—The rate of sulphur oxidation in soils increases with the fineness of the particles. During the oxidation process acidity is developed and calcium is freely brought into solution.

A. G. POLLARD.

Nitrification and denitrification in oxidising media. E. PARISI (Staz. sperim. agrar. ital., 1925, **58**, 449—472; Chem. Zentr., 1926, II., 102).—Nitrification in soils is not conditional on the presence of combined oxygen. In readily permeable soils, ammonia and nitrites are quantitatively converted into nitrates; in heavy soils, denitrification takes place and nitrogen is liberated. Probably the :CH·NH_2 group of amino-acids and amides reacts

with nitrous acid according to the equation $:CH{\cdot}NH_2+O{:}NOH=:CH{\cdot}OH+H_2O+N_2$. The oxygen consumption in a soil containing 15—20% of water is much greater than in a saturated soil Addition of sugar increases the oxygen consumption of saturated soils. C. T. GIMINGHAM.

Influence of lime and phosphatic fertilisers on the phosphorus content of the soil solution and of soil extracts. F. W. PARKER and J. W. TIDMORE (Soil Sci., 1926, **21**, 425—441).—The examination is recorded of displaced soil solutions and of dialysed aqueous soil extracts, of soils receiving various phosphatic fertiliser treatments. Liming increased the solubility of phosphate applied as superphosphate or basic slag; its effect on rock phosphates was small and uncertain, and with steamed bone flour a decided reduction in solubility resulted. The soils examined showed a marked ability to maintain a constant phosphate concentration in aqueous extracts. From theoretical considerations it is suggested that the plant absorbs some portion of its phosphate from the supposedly more concentrated layer of solution at the surface of the soil particles. The phosphate concentration in the displaced soil solutions was rarely greater than 1 pt. (PO_4) per million and frequently less than 0·1 p.p.m. A. G. POLLARD.

Chemical method of determining phosphoric acid requirements of agricultural soils. A. NEMEC (Compt. rend., 1926, **183**, 314—316).—0·005 mg. of phosphorus per 100 c.c. of solution may be determined colorimetrically by a method based on the formation of a blue coloration when phosphomolybdic acid is reduced by quinol in an acid medium, and treated with alkaline sodium sulphite. Comparison of results of determinations of soluble phosphoric acid by this method with the results of field trials on the same soils showed that those containing at least 35 mg. of phosphorus per kg. of dry soil require no superphosphate for cultivation of sugar-beet; others require treatment with fertiliser equivalent to 50 kg. P_2O_5 per hectare. For growing barley the minimum phosphorus content is 22 mg. per kg., for potatoes and meadow land 19 mg., and for oats 15 mg. L. F. HEWITT.

Synthetic calcium silicates as a source of agricultural lime. II. Comparison of their influence with that of other forms of lime, upon certain microbiological activities in the soil. R. M. BARNETTE (Soil Sci., 1926, **21**, 443—453; cf. B., 1925, 183).—Comparison was made of the effect of additions of calcium silicate, ground limestone, and calcium hydroxide on the bacterial numbers, rate of nitrification, and rate of sulphur oxidation in a number of soils. Slaked lime and the silicate produced similar typical increases in the numbers of bacteria, followed by decreases. The rates of denitrification following the various treatments differed according to the soil and the form of lime added, although the final yield of unleached nitrate was the same for the three treatments on any one soil. Similar results were obtained in a study of the increased rates of sulphur oxidation in acid soils following the application of the various forms of lime. A. G. POLLARD.

Conditions of the application and action of phosphates in black soil. III. Dynamics of the lime and ferric oxide plus alumina of the soil and the conditions influencing it. M. A. EGOROV and F. F. MACKOV (Ukraine Chem. J., 1926, **2**, Tech. part, 7—37).—Measurements have been made of the variations in the proportions of lime and of ferric oxide and alumina in black soil during the growth of crops and under the influence of different temperatures and moisture contents. By means of a special borer, the sample of soil, 20 cm. deep, was divided into a number of different layers, which were examined separately. The porosity of the soil amounts to 48·8—54·8% of the total volume occupied, the high values (up to 88·7%) obtained by other investigators by means of the pycnometric method being probably inaccurate. The amount of water-soluble material in the top layer (0—2 cm.) of the soil increased with the moisture content of this layer, but for the lowest layer (15—20 cm. down), which was always the wettest, the reverse was the case. In almost all seasons of the year the lime predominated over the ferric oxide and alumina in the extract of the soil, the amount of the last two oxides diminishing as the amount of the lime dissolved increased. The same relationship between the amounts of lime and ferric oxide dissolved on treatment with 10% hydrochloric acid solution was observed with black soil and forest loam. This determining influence of lime is confirmed by the variations of the quantity of this base in the aqueous extract of the soil at different periods of the day; the maximum, which is 1·7 times the minimum, occurred at the hottest part of the day (11 a.m.—1 p.m.) for the topmost layer and during the evening hours for the lowest layer examined. With high moisture content of the black soil (22%) the amount of lime passing into solution diminished as the temperature of storage of the sample was raised; for a constant temperature, the lime dissolved increased with the moisture content of the soil.
T. H. POPE.

Izum calcareous phosphorite and the plant under different conditions of soil moisture. M. A. EGOROV (Ukraine Chem. J., 1926, **2**, Tech. part, 40—49).—The fertilising value of this phosphorite is dependent in high degree on the moisture content of the soil, which must be about 80% of the maximum in order that the best results may be obtained from its use. T. H. POPE.

Iron phosphate as plant nutrient. F. MÜNTER (Z. Pflanz. Düng., 1926, **B5**, 305—311).—Vegetation experiments with wheat and red clover indicate that in neutral and alkaline soils the phosphorus in iron phosphate is less available to plants than that in superphosphate and basic slag. On the average, the efficiency of iron phosphate is about 50% of that of superphosphate.
C. T. GIMINGHAM.

Influence of boron on plants. A. CUSUMANO (Staz. sperim. agrar. ital., 1925, **58**, 440—448; Chem. Zentr., 1926, II., 102).—Boron acts as a stimulant to plants by inducing a better utilisation of nutrients. The optimum dose is 0·5 g. per sq. m. of soil surface. C. T. GIMINGHAM.

Manuring experiments with town sewage. W. ZIELSTORFF, A. KELLER, and E. SPUHRMANN (Z. Pflanz. Düng., 1926, B5, 289—304).—Observations upon crops irrigated with sewage led to experiments with town sewage with and without addition of effluent from cellulose factories. Town sewage alone was in no way harmful to mustard and oats grown in sand cultures; but addition of the cellulose effluent led to injury which was increased when the latter was used alone. The effluent from the cellulose factories was characterised by a high residue on evaporation. C. T. GIMINGHAM.

[Seed] stimulation and manuring. H. MÜLLER (Fortschr. Landw., 1926, **1**, 265—269; Chem. Zentr., 1926, II., 102).—The use of seed stimulants (solutions of magnesium chloride and of manganous and magnesium sulphates), combined with different types of manuring, has been tested on a variety of crops. No significant effects upon growth or yield were observed. The treatments did not increase the capacity of the plants to utilise either the soil nutrients or added mineral fertilisers. C. T. GIMINGHAM.

Effect of lime on preservation of the germinating power of seeds. Means of drying [seeds]. M. KONDO (Ber. Ohara Inst. landw. Forsch., 1926, **3**, 135—146, 147—151).—Experiments with a variety of seeds, extending over 11 years, show that storage in a cool place in air-tight vessels containing quicklime considerably increases the length of time during which the germinating power of the seeds is maintained. The effect is due to the drying action of the lime and is also observed when seeds are kept over sulphuric acid or calcium chloride. C. T. GIMINGHAM.

See also A., Aug., 869, *Azotobacter agile* (KOSTYTSCHEV, RYSKALTSCHUK, and SCHWEZOWA); **Relation of soil algæ to some soluble carbon compounds** (ROACH).

Silage experiments. ANNETT and AIYER.—See XIX.

Assimilation of carbon dioxide by plants. KÖGEL and STEIGMANN.—See XXI.

PATENTS.

Phosphate fertilisers. H. BLUMENBERG, JUN., Assr. to STOCKHOLDERS SYND. (U.S.P. 1,591,270-2, 6.7.26. Appl., [A] 12.5.25, [B] 23.6.25, and [C] 7.8.25).—(A) Ground phosphate rock is mixed with calcium nitrate in the presence of water and treated with sulphur dioxide, thus producing a fertilising material containing calcium nitrate and much soluble calcium phosphate, which is evaporated to dryness. Powdered quicklime is added to make a dry powder. (B) Ground phosphate rock is mixed with potassium sulphate, the mixture heated until fused, cooled, and ground. It is then treated with sulphur dioxide in the presence of water, potassium phosphate being formed. (C) Ground phosphate rock is mixed with ammonium sulphate, potassium chloride, and water in the presence of sulphur dioxide. C. T. GIMINGHAM.

Fertiliser. C. WITTS (Can. P. 253,880, 2.3.25).—Animal waste products, such as hair, wool, and leather are treated with water and steam until they contain 40% of water, and are then mixed with sodium hydrogen sulphate and cyanamide and again treated with steam. C. T. GIMINGHAM.

Production of solutions or suspensions of metal sulphides [plant sprays]. C. E. M. J. DE B. DE LAROCHE (F.P. 601,586, 15.7.25).—By using a solution of an alkali sulphide, solutions or stable suspensions of heavy metal (mercuric, iron) sulphides can be prepared, suitable for spraying plants. L. A. COLES.

XVII.—SUGARS; STARCHES; GUMS.

Crystallisation of sucrose solutions. H. I. WATERMAN and A. J. GENTIL (Chem. Weekblad, 1926, **23**, 345—438; cf. van Ginneken and Smit, B., 1919, 784 A).—The times required for crystallisation of supersaturated sucrose solutions at temperatures of 40°, 60°, 70°, and 90°, without seeding and after seeding with small (0·5—2·0 mg.) and large (150—200 mg.) crystals have been determined, and the results of van Ginneken and Smit confirmed. Crystallisation commences and proceeds most quickly after seeding with a large crystal, and the grain of the crystals formed is fine; seeding with a fine crystal causes slower crystallisation and coarser grain. It is emphasised that impurities in the solution exercise considerable effect. S. I. LEVY.

See also A., Aug., 823, *iso*Maltose (GEORG and PICTET); **Molecular unit of starch** (IRVINE and MACDONALD).

PATENTS.

Evaporation of sugar juices. A. R. VILA (U.S.P. 1,591,583, 6.7.26. Appl., 28.1.25. Conv., 13.1.25).—Defecated sugar juice is evaporated in a single-effect vacuum pan provided with an additional evaporating body, a forced circulation of the juice from the pan to the additional evaporator being produced and the liquor being heated in the latter by steam from the pan. The circulation continues until a mass at crystallising point is obtained. W. G. CAREY.

Treatment of molasses and sugary juices with a view to the recovery of the sugar. C. DEGUIDE (U.S.P. 1,579,090, 30.3.26. Appl., 3.11.25).—See E.P. 249,759; B., 1926, 561.

Treatment of the seed of the carob tree [to extract the gum]. J. F. A. AUDIBERT (U.S.P. 1,593,544, 20.7.26. Appl., 12.10.25. Conv., 10.10.24).—See E.P. 241,186; B., 1926, 642.

XVIII.—FERMENTATION INDUSTRIES.

Determination of added water in wines. C. A. Grau (Rev. Fac. Cien. Quím., 1925, 3, 133—142) —Genuine wines do not contain nitrates, whilst water used for dilution may contain nitrates; hence the detection of nitrates in wines may furnish information as to this type of sophistication. The test for nitrate is made by means of the diphenylamine reaction on the liquid obtained after removal of alcohol by distillation and defecation of the residue by means of basic lead acetate and animal charcoal. Excess of diphenylamine should be avoided. The reagent recommended is prepared by adding 15 c.c. of strong sulphuric acid to 2—3 mg. of commercial diphenylamine, diluting with 6 c.c. of water to obtain complete solution, and making up the volume to 100 c.c. with strong sulphuric acid.

G. W. Robinson.

See also A., Aug., 864, **Affinity constants of enzymes** (von Euler); **Complement of amylase** (Pringsheim and Otto). 865, **Hydrogenase** (Grüss); **Invertase; Affinity of invertase. Effect of acidity on enzymic breakdown of sucrose; Affinity groupings of invertase** (von Euler and Josephson); **Affinity of invertase for different sugars** (Josephson); **Hydrolysis of sucrose by invertase in presence of α-methylglucoside** (Nelson and Post); **Specificity of disaccharases. Taka-maltase and taka-invertase** (Leibowitz and Mechlinski); **Enzymes contained in taka-diastase preparations** (Sano). 867, **Buffer capacity and biochemical action** (Moser); **Method of arresting fermentation, particularly in the case of liquids containing alcohol and sugar, and of rendering such liquids non-fermentable** (Boulard); **Effect of temperature on growth of yeast in various media** (Sherwood and Fulmer). 868, **Reductase of yeast. Purification of co-reductase** (von Euler and Nilsson); **Lactic acid fermentation** (Virtanen and Karström); **Production of lactic acid from sugar by methylglyoxal-dismuting bacteria** (Neuberg and Gorr).

Patents.

Denatured alcohol. E. M. Flores (U.S.P. 1,591,084, 6.7.26. Appl., 9.6.22. Conv., 13.6.21).— To crude ethyl alcohol, is added 1% of crude wood alcohol containing acetone, and 1% of a light fraction of mineral oil distilling between 40° and 150°.

W. G. Carey.

XIX.—FOODS.

Refrigeration of meat. II. Freezing of beef and mutton press juices. J. R. Vickery (Aust. J. exp. Biol., 1926, 3, 81—88; cf. B., 1926, 604).— Examination of expressed beef and mutton juices before freezing and after thawing indicated that a fairly complete reconstitution of the juice was attained when the rate of freezing was such as to allow of a latent period up to 3 hrs. Slow thawing favoured this reconstitution. Ultramicroscopical examination showed that freezing and thawing reduced the number of single colloidal particles in the juice to an extent which increased as the rate of freezing decreased. This effect was scarcely appreciable in the case of mutton juice. It is suggested that alterations in beef juice during freezing and thawing are mainly dependent on changes in the sarcolemma and that differences in the properties of beef and mutton under these conditions are the outcome of inherent differences in the respective sarcolemma.

A. G. Pollard.

Determination of creatinine in soup preparations. W. Müller (Mitt. Lebensm. Hyg., 1926, 17, 45—51; Chem. Zentr., 1926, II., 503).—The process of Sudendorf and Lahrmann (B., 1915, 1027) is recommended for the determination of the total creatinine in meat soup cubes. The somewhat simpler process of Folin and Geret described in Swiss text-books often gives too high results, as it does not involve the separation of all the compounds which may be mistaken for creatinine.

B. W. Clarke.

Freezing point of milk from diseased cows. J. Straub (Chem. Weekblad, 1926, 23, 338—340). —The depression of the freezing point for milk is caused by (*a*) soluble chloride, (*b*) sugar, and (*c*) other solids. The depression due to (*c*) is about 0·14° for milk from healthy cows; for milk from cows with inflamed udders and for colostrum it is from 0·16° to 0·20° or more. The depression 0·14° for sound milk corresponds exactly with the content of soluble phosphates and citrates; the greater depressions for other milks have not yet been explained.

S. I. Levy.

Application of Gerber's process to the determination of fat in cocoa and chocolate. J. Ruffy (Ann. Chim. Analyt., 1926, 8, 225—227). —The fat in 3·5 g. of the substance is extracted by shaking with 20 c.c. of ether, the solution is centrifuged, and 10 c.c. are added to 13 c.c. of strong sulphuric acid in a butyrometer. The ether is dissolved by the sulphuric acid, and after centrifuging, the fat is separated and its volume read, the percentage of fat being obtained by dividing by two. The process is as accurate as the usual methods. B. W. Clarke.

Disintegration of rice straw [to increase its fodder value]. H. Iwata (J. Dept. Agric. Kyushu, 1926, 1, 217—240).—The effect of various treatments on the incrusting substances of rice straw and on its starch value and digestibility, as determined by feeding trials, has been investigated. Boiling for 3 hrs. with water, or digestion for 4 hrs. with a cold 0·25% solution of sodium hydroxide had little effect. Treatment with stronger solutions of sodium hydroxide (0·75% or 1·5%), or boiling with milk of lime (1% CaO) for 3 hrs. caused considerable disintegration, reduced the content of lignin, and considerably increased the starch value, the amount of digestible matter, and the palatability to animals.

C. T. Gimingham.

Silage experiments at Nagpur. H. E. ANNETT and A. R. P. AIYER (Mem. Dep. Agric. India, 1926, 8, 189—209).—Changes in composition taking place during the conversion of green *juar* fodder (*Sorghum vulgare*) into silage have been studied. The total change in weight did not amount to more than 2—3%. There was no diminution in the proportion of pure to crude protein as found by Amos and Woodman in the case of silage made from oats and tares (cf. B., 1923, 115 A). The ratio of amino-acids to volatile bases was low. No significant differences were observed between silage made in pits dug in the ground and in the below ground part of a tower silo; in the upper portion of the silo the temperature became very high and caramelisation took place.
C. T. GIMINGHAM.

See also A., Aug., 821, **Constitution of pectin** (AHMANN and HOOKER). 858, **Stability of evaporated milk during sterilisation** (BENTON and ALBERY). 866, **Rennin. Effect of previous heating on coagulability of caseinogen. Replacement of calcium by other cations. Replacement of phosphate by other substances** (MARUI). 871, **Colour reactions attributed to vitamin-A** (CARR and PRICE); **Antirachitic properties of irradiated sterols** (ROSENHEIM and WEBSTER); **Antirachitic factor of cod-liver oil** (LESNÉ and SIMON); **Influence of diet and sunlight on amount of vitamin-A and vitamin-D in milk afforded by a cow** (CHICK and ROSCOE). 872, **Vitamins** (VON EULER and LINDSTAL); **Interrelation of vitamin effects** (VON EULER and WIDELL); **Inequality in content of water-soluble vitamin-B of yeast extracts of different origin; Is the water-soluble vitamin-B of brewer's yeast derived from the culture medium?** (RANDOIN and LECOQ); **Vitamins-A, -B, and -C of papaya** (MILLER); **Action of reducing agents on antiscorbutic factor inactivated by aeration** (DAUBNEY and ZILVA); **Bezssonoff's reagent for vitamin-C** (LOEWY); **Accessory factors for growth** (VON EULER and RYDBOM).

PATENTS.

Increasing the nutritive value of food. J. F. and G. DAUBEK (E.P. 254,388, 1.4.25).—Fodder and foodstuffs containing material difficult of assimilation, such as husks, are treated with yeast (torulæ) cultivated, preferably in association with suitable bacteria, on pentosans. The mixture of yeast and food to be treated is placed in a jigging apparatus and warmed to 50° when the proteins not responsive to the action of gastric juice are converted into a more digestible condition. The process is also suitable for eliminating the smell from slaughter-house refuse, or for stabilising the lipases in oleaginous fruits, such as pressed olives, and thus preventing the unpleasant smell from the enzymes present. W. G. CAREY.

Condensed milk and method of making it. J. R. HATMAKER (E.P. 254,941, 5.11.25).—Condensed milk of superior taste and colour to that prepared by the usual method is obtained by rapidly drying a portion of the fresh milk, and then incorporating the freshly-dried milk solids by means of a homogeniser with the remainder of the milk warmed to 40—50°. W. G. CAREY.

Ripening and preservation of citrus fruits. A. HOOPER (Austral. P. 17,327, 14.4.24).—The fruits are treated in an airtight chamber with a gaseous mixture of ethylene and formaldehyde.
B. W. CLARKE.

Pectin from apples. R. PAUL (F.P. 602,336, 21.8.25).—Apples are pulped by heating with slightly acidified water for 12 hrs. at 100°, then cooked under pressure at 115—120°, cooled rapidly, then cooked again, and centrifuged out of contact with the air.
B. W. CLARKE.

Recovery of alcohol in bakeries. M. ANDRUSIANI (F.P. 603,218, 18.9.25).—The vapour from the ovens in a bakery is passed through a fractional condenser, where water vapour is removed, and then through a further condenser where alcohol is condensed and recovered. B. W. CLARKE.

Preservation of eggs. E. MERCK (F.P. 603,312, 13.8.25. Conv., 19.8.24).—Eggs are treated with solutions of primary metals or alkaline-earth phosphates or magnesium phosphate, or with solutions of secondary phosphates in phosphoric acid or with free phosphoric acid. A preliminary exposure in a rarefied atmosphere may be given. R. BRIGHTMAN.

Ascertaining the moisture content of cereals and the like. H. J. DENHAM and G. WATTS, Assrs. to H. SIMON, LTD. (U.S.P. 1,593,161, 20.7.26. Appl., 6.6.25. Conv., 30.6.24).—See E.P. 239,637; B., 1925, 939.

Purification of industrial effluents (F.P. 597,515).—See XXIII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Loss of morphine in powdered opium by keeping. A. C. ABRAHAM and J. RAE (Pharm. J., 1926, 117, 3—5, 32—34).—Experiments on the loss of morphine in powdered opium on keeping in air, oxygen, and carbon dioxide, and after heating at 98° indicate that opium contains a peroxydase (opiase) to which are due the changes in the morphine content of powdered opium. The enzyme is destroyed in a powder containing 1—2% of water, at 98°. A modification of the method of determining morphine in opium is discussed. B. FULLMAN.

Utilisation of unripe fallen "ponkan" fruits. K. KAFUKU and C. HATA (J. Soc. Chem. Ind. Japan, 1926, 29, 212—214).—Fallen unripe fruits of the "ponkan" or sweet mandarin of Formosa, consist of about 13·5% of peel and 86·5% of pulp by weight. From the pulp, more than 50% of the juice can be pressed out containing about 5·7% of calcium citrate. The juice contains, in addition to citric acid, 4—8% of invert sugar; after removal of the citrate the

juice can be fermented, and distilled to obtain an alcoholic beverage. The peel, when distilled with steam, gives about 1·7% of a fragrant oil having the following characteristics:—d^{15} 0·8422, n^{30}_D 1·4687, α+89·12°, specific viscosity 1·002, acid value 0·6, ester value 5·3, ester value after acetylation 46·1, total aldehyde 2%. The oil contains limonene and an aldehyde which gives an oxime of m.p. 76—78°,

K. KASHIMA.

Determination of the anthraquinone derivatives in cathartic drugs. G. D. BEAL and M. C. T. KATTI (J. Amer. Pharm. Assoc., 1925, **14**, 865—873).—Dael's method (Bull. Acad. roy. med. Belg., 1921, **1**, 185) is preferred. Details are given of the application of the method to rhubarb, cascara, senna, frangula, and pure emodin. Colorimetric methods are inaccurate unless the colour standards are prepared from the anthraquinone extract with the greatest care and from the species under examination.

A. A. ELDRIDGE.

Neosalvarsan. I. Analysis. L. FREEDMAN (J. Lab. Clin. Med., 1926, **11**, 528—541).—A reversible reaction takes place when arseno-compounds are titrated with iodine in the presence of a mineral or organic acid, the extent being proportional to the acidity. The combined formaldehyde and sulphoxylate group is oxidised in neutral solution by 2 I to the formaldehyde hydrogen sulphite group; in the presence of dilute hydrochloric acid it is hydrolysed, 4 I being then required for complete oxidation.

A. A. ELDRIDGE.

Detection of vanillin. Sensitivity of the nitric acid test. J. MCLANG (Chem. Trade J., 1926, **78**, 73—74).—The ferric chloride test for vanillin cannot be used at dilutions greater than 1 pt. of vanillin in 1300 of water. By mixing 2 vols. of vanillin solution with 1 vol. of nitric acid (d 1·35) and heating for 35—60 sec., a definite yellow coloration is obtained with vanillin solutions of a dilution of 1 pt. in 128,000 pts. of water. The limiting sensitivity of the reaction appears to be 1 pt. of vanillin in 340,000 pts. of water when the coloration produced after heating for 1 min. is examined by transmitted light, and 1 pt. in 500,000 when reflected light is used against a white background. The test is serviceable even with coloured or impure liquors if the test sample is given a preliminary treatment with diamantin or other decolorising or clarifying agents.

R. BRIGHTMAN.

Assay of salicylates and benzoates. A. H. CLARK (J. Amer. Pharm. Assoc., 1926, **15**, 6—11).—For the assay of sodium salicylate and sodium benzoate determination of the metal as chloride is preferred; alternatively, and more generally, the acid is weighed after liberation and extraction with chloroform. Ignition, followed by titration of the carbonate, and volumetric absorption of bromine or iodine are rejected.

A. A. ELDRIDGE.

Catalytic oxidation of organic compounds in the vapour phase. C. R. DOWNS (J.S.C.I., 1926, **45**, 188—193 T).—Catalytic processes of partial oxidation, *e.g.*, naphthalene to phthalic anhydride, depend almost entirely for their success on accurate temperature control. A converter tube designed to secure uniformity of temperature consists of a U-tube of thin sheet steel containing the catalyst deposited on grained aluminium with untreated aluminium as a preheater. The tube is immersed in a bath of molten lead. An improvement is produced by coating the catalyst directly on the tube walls. Even in this case the maximum temperature is at the centre of the tube at the inlet end and the diameter of the tube is therefore of importance. A more perfect arrangement consists of a nest of tubes with jacket preheaters immersed in mercury or sulphur boiling under any desired pressure. The reaction is started electrically and maintains itself. A converter of this type, 3 ft. diameter × 4 ft. high, will produce 1 ton of phthalic anhydride per 24 hrs. Certain forms of vanadium oxide used as catalyst functioned satisfactorily for six months without attention and a yield of 80 lb. of phthalic anhydride per 100 lb. of naphthalene was obtained. Other compounds which can be prepared commercially in a similar way are benzaldehyde and benzoic acid from toluene, anthraquinone from crude anthracene, maleic acid convertible into malic acid, and fumaric and succinic acids.

C. IRWIN.

Synthetic manufacture of alcohol and acetic acid [and acetone]. P. PASCAL (Mém. Poudres, 1926, **22**, 1—30).—The synthetic production of acetic acid and alcohol from acetylene comprises three stages, viz., conversion of acetylene into acetaldehyde, reduction of the aldehyde to ethyl alcohol, and oxidation to acetic acid. Acetaldehyde is obtained by passing a rapid current of acetylene into a solution of a mercury salt dissolved in sulphuric or acetic acids to which ferric sulphate has been added to prevent precipitation of mercury. The reaction mixture should be heated, but its temperature need not exceed 65°. The strength of sulphuric acid recommended is 20%, with 2% of mercury salt. When a ternary catalyst containing mercury, iron, and vanadium in the atomic proportions 2 : 2 : 0·2 was used, the total yield of acetaldehyde was 10·5% and the ratio aldehyde : mercury was 9·2. When the acetaldehyde was removed from the excess acetylene in a packed column and not in bubblers these figures become 20·5 and 14·6 respectively. The conversion of aldehyde into alcohol was effected by electrolytic reduction in a diaphragm cell with a cathode of pure lead, the current required being 2—3 amp. per sq. dm. The cell charge should be 10% sulphuric acid to which 10% of aldehyde is added. Subsequent additions of aldehyde may be increased to 30%. The temperature in the cell should not exceed 40°. Under these conditions any acetal formed was converted into acetic acid. The yield of alcohol reached 96% and the current efficiency always exceeded 92%. The oxidation of aldehyde to acetic acid was studied only on a laboratory scale. The catalyst used was potassium permanganate (0·3%) and the temperature

of the reaction was kept between 50° and 55°. Acetic acid was converted into acetone by passing its vapour over manganous oxide deposited on pumice. The catalyst was contained in an aluminium tube heated to 450°. Natural pyrolusite or Indian manganese ore were also effective catalysts. A production of 20 metric tons per cub. m. of catalyst could be obtained without exhausting the catalyst. By refrigerating the vapours from the catalyst, 94·7% of the acetone formed could be recovered.

S. BINNING.

Determination of camphor. O. ASCHAN (Finska Apoth. Tidsk., 1925, 49).—One gram of the substance is triturated with 2 c.c. of glacial acetic acid, 1 g. of semicarbazide hydrochloride, and 1·5 g. of freshly fused potassium acetate, the mixture heated in a plugged tube at 70° for 3 hrs., stirred after cooling with 10—15 c.c. of water, the semicarbazone collected on a filter, washed with water, dried in air, and finally washed with light petroleum.

A. A. ELDRIDGE.

Determination of iodine in organic combination. C. W. GEITER (Amer. J. Pharm., 1926, 98, 352—355).—Organically combined iodine in high-percentage iodine compounds is determined by heating with potassium carbonate until the mass is decarbonised, oxidising the iodide formed to iodate by means of sodium hypochlorite and phosphoric acid, and determining the iodate volumetrically in the usual way. By this method thymol iodide shows an average content of 46·1% of iodine, results by the present U.S.P. method (oxidation with permanganate and sulphuric acid) being 0·42% lower.

B. FULLMAN.

Pyrogenic oxidation of turpentine in presence of contact copper [copper gauze]. E. I. ORLOV (Ukraine Chem. J., 1926, 2, ii., 1—6).—The pyrogenic oxidation of Russian turpentine in presence of heated copper gauze yields as much as 60—70% of oily products which, when distilled under a dephlegmator, give a fraction, b.p. 155—183°, having a pleasant smell. This fraction may be used in the preparation of perfumes as a solvent for essential oils.

T. H. POPE.

Reaction to distinguish between anise oil and star-anise oil. W. P. H. VAN DEN DRIESSEN MAREEUW (Pharm. Weekblad, 1926, 63, 929—934).—After examination of tests depending on the presence (in star-anise oil) of cineole, safrole, and sesquiterpenes (absent in anise oil), a satisfactory method was based on the differences in the nitrosites formed by the two oils. The crystals formed on keeping after treatment with sodium nitrite and acetic acid were found to melt at 104—105° and to give a purple ring on treatment with sulphuric acid in glacial acetic acid solution in the case of star-anise oil; those obtained from anise oil melted at 109—110° and gave a reddish-brown colour.

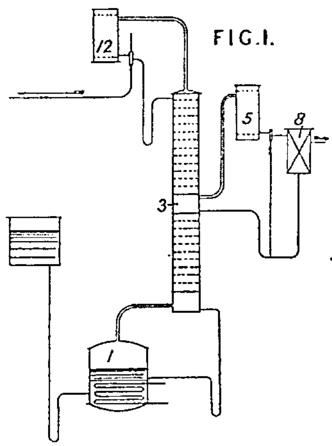
S. I. LEVY.

See also A., Aug., 827, **Synthesis of lecithin** (GRÜN and LIMPÄCHER). 830, **α-Naphthylcarbimide as reagent for phenols and aromatic amines** (FRENCH and WIRTEL). 834, **Chemical constitution, physiological action, and physical properties in a series of alkyl *p*-aminobenzoates** (ADAMS, RIDEAL, BURNETT, JENKINS, and DREGER). 840, **Natural musk** (WALBAUM). 850, **Characteristic reactions for strychnine, morphine, codeine, and formaldehyde** (ALOY, VALDIGUIE, and HOY). 851, **Behaviour of arsenobenzenes towards molecular oxygen. I. II.** (MASCHMANN). 852, **Mercury compounds of "medinal" and test for identifying "medinal"** (RUPP and MÜLLER). 853, **Detection of minute quantities of formaldehyde** (SABALITSCHKA and HARNISCH); **Reaction to distinguish between cocaine and novocaine** (GERHARDT). 863, **Substances in ergot affecting the uterus** (FORST); **Narcotic action of pure ether** (NIJK). 869, **General properties of cryptotoxins; tetanus cryptotoxin** (VINCENT).

PATENTS.

Manufacture of simple or mixed ethers of the fatty series. DISTILLERIES DES DEUX-SÈVRES (E.P. 243,736, 25.11.25. Conv., 26.11.24).*—The water

FIG. 1.



formed in the preparation of simple or mixed aliphatic ethers by reaction between alcohols and sulphuric acid, phosphoric acid, benzenesulphonic acid, etc., is removed by conducting the operation in the presence of a substance, *e.g.*, a liquid insoluble or only slightly soluble in water which produces a mixture of minimum b.p., rich in water. For the preparation of ethyl ether, a 20 : 15 : 1 mixture of ethyl alcohol, sulphuric acid, and petrol of b.p. 100° is heated in the boiler, 1, and the vapours of ethyl ether, water, and petrol pass to a rectifying column, 3, to which a condenser, 5, and dehydrating apparatus, 8, are connected. The ether is removed by a condenser connected to the upper part of the column. In a modification for preparing highers ethers, the ethers are removed from the bottom of the rectifying column. In the case of butyl ether the products of reaction themselves form a mixture of minimum b.p. with water without any added substance.

B. FULLMAN.

Manufacture of a preparation which emits β-rays. M. WRESCHNER and L. F. LOEB (E.P.

[A] 248,765 and [B] 255,000, 5.3.26. Conv., [A] 6.3.25, [B] 21.8.25).—(A) A new preparation emitting β-rays which is insoluble in the fluids of the body and is suitable for treatment of cancer is obtained by treating a solution of a uranium salt containing a ferric salt with sufficient ammonium carbonate to precipitate the iron as hydroxide, together with the adherent uranium-*X*, whilst the uranium remains in solution. The precipitated ferric hydroxide, which absorbs uranium-*X* quantitatively, is thoroughly washed and suspended in a fluid isotonic with blood serum, for instance, in a physiological solution of common salt. (B) The product obtained as described under (A) is dissolved in a suitable acid (preferably hydrochloric acid) and after boiling the solution is neutralised and again treated with an excess of ammonium carbonate. By repeating this procedure one or more times a very pure and less toxic preparation is obtained. The preparation thus obtained, or that obtained by process (A) may be suspended in a solution of a suitable sugar, for instance, sucrose, which is not attacked by the glycolytic ferments present in the cancer tissue, or in an oil, in place of an isotonic liquid. R. BRIGHTMAN.

Manufacture of new salts of cinchona alkaloids. ETABL. POULENC FRÈRES (E.P. 249,849, 16.3.26. Conv., 28.3.25).—The *acetamidohydroxyphenylarsinates* of the cinchona alkaloids are prepared by reaction between the pure or mixed bases and the free acetamidohydroxyphenylarsinic acids or between the pure or mixed alkaloid salts and a salt of the arsinic acid. The products are but slightly soluble and are antiseptics. Details are given of the preparation of the *3-acetamido-4-hydroxyphenylarsinates* of quinine and quinidine. B. FULLMAN.

Production of new ester mixtures. W. CLAASEN (E.P. 250,910, 23.2.26. Conv., 14.4.25).—The products obtained by esterifying with aliphatic, aromatic, or cyclic alcohols the mixture of α- and β-methyladipic acids and their homologues formed by oxidation of the methyl*cyclo*hexanols obtained by the catalytic reduction of crude cresols, may be used as camphor substitutes in the preparation of celluloid. The mixed ethyl esters have b.p. 124—150°/12 mm., and the mixed *cyclo*hexyl esters b.p. 214—241°/9 mm. B. FULLMAN.

Production of diaminodiarylurea [diaminodiarylcarbamide] or its derivatives. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 254,667, 21.5.26).—4 : 4′-Diamino-*s*-diphenylcarbamide and its symmetrically nuclear-disubstituted derivatives are prepared by heating carbamide with *p*-phenylenediamine or its nuclear-substituted derivatives. 4:4′-Diamino-*s*-diphenylcarbamide is obtained from carbamide and *p*-phenylenediamine at 110—120°, or from carbamide and *p*-phenylenediamine in the presence of *o*-dichlorobenzene at 130°, and 4 : 4′-diamino-3 : 3′-dimethyl-*s*-diphenylcarbamide from carbamide and 2 : 5-tolylenediamine in the presence of trichlorobenzene at 120—130°. B. FULLMAN.

Extraction of the essential principles of plants, flowers, fruit, animal matter, etc. E. FORAY (E.P. 254,758, 9.1.25).—The essential principle is extracted from the raw material with alcohol or ether (or with water, the product being subsequently transferred to alcohol or ether) and deposited on a substance such as sodium chloride, sugar, sago, flour, etc., by evaporation or centrifuging. For example, 1 kg. of camomile blossom is treated with 100 g. of water at 30° for half-an-hour. 1 kg. of water at 80° is added, and then 500 g. of well-cooled ether in small quantities. The liquid is kept at 80° for 1 hr., and the ethereal solution then evaporated in the cold or *in vacuo* on 250 g. of finely divided sugar. The products keep well and may be utilised as such or treated with a solvent to recover the perfume, etc. B. FULLMAN.

Manufacture of oxygenated organic compounds. J. Y. JOHNSON. From BADISCHE ANILIN & SODA FABR. (E.P. 254,760, 2.2.25).—The production of methyl alcohol and other oxygenated organic compounds from oxides of carbon and hydrogen is catalysed by iron, nickel, or cobalt in chemical combination or in the form of alloys or solid solutions, but not combined with copper or silver alone. The catalysts must be stable under the working conditions employed (200—600°), and must not yield metallic iron under such conditions either by reduction or by decomposition. Suitable contact masses may consist of an oxide or other compound of iron, nickel, or cobalt in conjunction with a difficultly reducible compound, *e.g.*, oxide of chromium, vanadium, tungsten, zirconium, aluminium, titanium. Alternatively the metals may be used as an alloy with one or more suitable metals such as manganese, chromium, tungsten, tin, zinc, and the like, in sufficient quantity to leave no free iron and the like present, or in the state of a compound with one or several metalloids, other than carbon, such as silicon, boron, sulphur, phosphorus, or arsenic. The gases must be free from volatile compounds of iron, nickel, and cobalt, and the surface of the apparatus exposed to the gases must likewise be free from these metals. Part, or in the case of carbon monoxide, even all of the hydrogen may be replaced by water vapour or by hydrocarbons such as methane. With an increased percentage of carbon monoxide the proportion of higher molecular products is generally increased, and formation of water decreased, whereas the relative production of methyl alcohol is increased by a preponderance of hydrogen. The efficiency of the catalysts is increased by the addition of an alkali or an alkali compound. In many cases the products differ more or less from those obtained with catalysts free from iron, nickel, or cobalt, the new catalysts yielding larger amounts of higher alcohols, ketones, and acids. Several examples of the preparation and application of the catalysts are given. R. BRIGHTMAN.

Manufacture of diarylguanidines. BRIT. DYESTUFFS CORP., LTD., C. J. T. CRONSHAW, and W. J. S. NAUNTON (E.P. 255,220, 4.7.25).—Aniline or a homologue is added slowly to a cold well stirred mixture of liquid cyanogen chloride or bromide and water, and stirring continued till the formation

of the cyanoanilide or cyanoarylide is complete. The temperature is then raised to about 90° for 4 hrs., when a clear solution of diarylguanidine hydrochloride is formed, from which the base is isolated in almost theoretical yield by running into hot caustic soda solution. A. DAVIDSON.

Obtaining perfumes from flowers and other parts of plants. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 255,346, 4.2.26).—Air or an indifferent gas is passed over flowers etc., suitably heated, and the perfume then absorbed from the gas in absorption carbon or silica gel; or the flowers are extracted with a solvent and the perfume absorbed from the solution by the absorption medium. A. DAVIDSON.

Partially oxidising gaseous hydrocarbons. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,588,836, 15.6.26. Appl., 26.2.24).—Methane mixed with 1—3·5 vols. of air is passed at 550° over a catalyst consisting of molybdic oxide or an oxide or compound of a high-melting, electronegative metal of low atomic volume, *e.g.*, vanadium, manganese, tungsten, uranium, chromium, titanium, or zirconium, or of a mixture of such compounds, preferably with addition of another metal oxide, such as silver oxide; the period of contact with the catalyst is about 1 sec. Formaldehyde and formic acid are produced, the yield of the former being 6·75% on the methane. It is of advantage to employ a large number of catalyst chambers in series, the products of oxidation being removed between successive chambers. The air may be ozonised. The process is applicable to methane homologues, to coal gas, and to petroleum still gas, the products in the last case consisting of a mixture of aldehydes and acids. T. S. WHEELER.

Production of formamide and hydrocyanic acid whether free or in the form of ammonium cyanide. J. Y. JOHNSON. From BADISCHE ANILIN & SODA FABR. (E.P. 254,787, 6.4.25).—By heating alkyl formate vapour (sometimes mixed with formamide, ammonium formate, or alcohol) and ammonia in the presence of a dehydrating contact mass, such as alumina, thoria, aluminium phosphate, zeolites, silica gel, etc., there is produced either formamide (high speed of gas current and/or low temperature), or hydrocyanic acid (low speed of gas current and/or high temperature), or both together. An amount of ammonia exceeding that calculated from the equation $H{\cdot}CO_2R+NH_3=H{\cdot}CONH_2+ROH$ or $HCN+ROH+H_2O$ often increases the yield, as does the presence of inert gases. The conditions of the reaction also depend upon the apparatus and the heating of the catalyst. For example, a mixture of 2 litres of methyl formate vapour and 80 litres of ammonia gas per hour passed over 50 c.c. of precipitated alumina heated to 260° yields 95% of the theoretical quantity of hydrocyanic acid as ammonium cyanide. At 100 times this speed, or at lower temperatures (*e.g.*, 180°), or with much less ammonia (*e.g.*, 2 litres of methyl formate vapour and 4 litres of ammonia gas) the main product is formamide. B. FULLMAN.

Process of making crotonaldehyde. F. W. LOMMEN, Assr. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,587,661, 8.6.26. Appl., 24.8.25).—Aldol is added slowly to a boiling 5% solution of sulphuric or phosphoric acid so that the water and crotonaldehyde, which are produced, the latter in over 90% yield, distil off through a rectifying column as formed. It is preferable to carry out the reaction at reduced pressure. T. S. WHEELER.

Hydroxybenzoylaminobenzenearsonic acids. I. G. FARBENIND. A.-G., Assees. of L. BENDA (U.S.P. 1,588,381, 8.6.26. Appl., 2.6.24. Conv., 14.6.23).—Arsinic acids of *o*-aminophenols yield on treatment with benzoyl chloride in presence of caustic alkali the monobenzamido-compounds exclusively, no substitution occurring in the hydroxyl group. The products are relatively non-toxic and are of therapeutic and prophylactic value in respect to trypanosomes and spirochaetæ. They may be administered *per os* or parenterally. The benzoylation of *p*-amino-*m*-hydroxybenzenearsinic acid (cf. Benda, A., 1912, i., 148) and of *m*-amino-*p*-hydroxybenzenearsinic acid is described. T. S. WHEELER.

Preparation of derivatives of aromatic arseno-compounds and their complex metal compounds. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of L. LÄUTENSCHLÄGER, K. STREITWOLF, and A. FEHRLE (G.P. 423,036, 17.4.23).—Acid radicals are introduced into the amino-groups of 3 : 3′-bismethyl- or ethyl-amino-4 : 4′-dihydroxyarsenobenzene and if desired the products are treated with metal oxides or salts. Alternatively the parent amino-compound is treated first with the metal oxide or salt and acid radicals are then introduced into the amino-groups if desired. Both series of reactions yield products of therapeutic value. Similar compounds are also obtained by treating the derivatives of bisalkylaminodihydroxyarsenobenzene, obtained by introducing acid radicals into the amino-groups, with metal oxide or salt derivatives of the parent amino-compound or of the products obtained by introducing acid radicals into the amino-groups. For example, bismethylaminodihydroxyarsenobenzene hydrochloride is heated for 30 min. at 55° in aqueous alkali with formaldehydesulphoxylate, and the filtered solution is treated with carbon dioxide until neutral to phenolphthalein, poured into alcohol, and the sodium sulphoxylate derivative of the parent substance precipitated by addition of ether. It is soluble in physiological salt solution. Formaldehyde and sulphurous acid yield with the parent substance the monoformaldehyde hydrogen sulphite compound of 3 : 3′-bismethylamino-4 : 4′-dihydroxyarsenobenzene, which on further treatment with sulphurous acid and formaldehyde gives the diformaldehyde hydrogen sulphite compound. The hydrochloride of the parent substance gives with silver nitrate a complex silver compound which is isolated as the sodium salt. By the action of formaldehyde sodium hydrogen sulphite on the complex silver compound its

N-methylsulphurous acid derivative is obtained This may be combined with bismethylaminodihydroxyarsenobenzene formaldehydesulphoxylate. Bismethylaminodihydroxyarsenobenzene condensed with chloroacetic acid gives the *N*-acetic acid derivative. 3-Ethylamino-4-hydroxybenzene-1-arsinic acid (decomp. 270°), on reduction yields 3 : 3′-bismethylamino-4 : 4′-dihydroxyarsenobenzene, which gives similar compounds to the methyl homologue.

T. S. WHEELER.

Benzoylecgonine ester. E. POULSON (U.S.P. 1,588,466, 15.6.26. Appl., 12.5.24. Conv., 15.5.23).—Benzoylecgonine ester derivatives in which the ester-forming group is an unsaturated alkyl or an aryl radical are of therapeutic value as local anæsthetics both *per se* and in the form of their salts. They are prepared by Merck's method (A., 1886, 163) by heating benzoylecgonine with a halide of the radical it is required to introduce. For example, benzoylecgonine heated under pressure with benzyl chloride at 110° gives *benzoylbenzylecgonine hydrochloride*, m.p. 123° in 80% yield; the free *base* forms an oil which in absolute alcohol has $[\alpha]_D = -26{\cdot}97°$. *Benzoylallylecgonine hydriodide* has m.p. 180°; the free base has m.p. 92° and in absolute alcohol has $[\alpha]_D = -31{\cdot}00°$; the *hydrochloride* has m.p. 176° and in absolute alcohol $[\alpha]_D = -37{\cdot}30°$.

T. S. WHEELER.

Pharmaceutical product. A.-G. FÜR ANILIN-FABR., Assees. of H. LANGER (U.S.P. 1,588,753, 15.6.26. Appl., 17.6.24. Conv., 20.10.23).—Mixtures of hexamethylenetetramine with 1—4 pts. of the hydrohalide of an aminofatty acid or of a derivative such as a lactam or ester are of therapeutic value as internal disinfectants for the urinary organs, and can be stored dry for long periods without deterioration. Suitable compounds for mixing with hexamethylenetetramine comprise alanine hydrochloride and betaine hydrochloride and hydrobromide.

T. S. WHEELER.

Manufacturing thiazoles. L. B. SEBRELL and C. W. BEDFORD, Assees. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,591,440, 6.7.26. Appl., 12.4.24).—Arylthiazoles are prepared by heating aryl-substituted thiocarbamides in presence of sulphur.

S. I. LEVY.

Preparation of nitrophenol alkyl ethers. VEREIN FÜR CHEM. U. METALL. PRODUKTION (F.P. 602,977, 7.9.25).—Chloronitro-substitution products of the aromatic series are heated with alcohols and alkali hydroxides in presence of a complex compound of copper oxide and a polyhydric alcohol at ordinary or increased pressures to give the corresponding nitrophenol alkyl ethers in satisfactory yield, the formation of dihalogenazo- and -azoxy-compounds being almost completely suppressed. For example, cuprous chloride, glycerol, sodium hydroxide, and 1 : 4-dichloro-2-nitrobenzene are heated in methyl alcohol under a reflux to give a chloronitroanisole in almost theoretical yield. *p*-Chloronitrobenzene heated with copper sulphate or chloride, glycerol, sodium hydroxide, and ethyl alcohol at 100° in an iron autoclave gives directly *p*-nitrophenetole, f.p. 56—57°, and small quantities of *p*-nitrophenol.

T. S. WHEELER.

Preparation of complex metal-gelatoses. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of W. SCHOELLER and R. DIRKSEN (G.P. 423,080, 2.4.24).—By mixing a solution of gelatose, slightly alkaline with sodium hydroxide, with a concentrated aqueous solution of silver nitrate and a salt of another metal with chemotherapeutic properties, and evaporating to dryness in a vacuum, binary metal-gelatoses are obtained which have greater therapeutic powers than the simple metal-gelatoses, and less pronounced secondary actions. They contain about 5% of silver and 5% of the other metal. In the *silver-zinc-gelatose* (yellowish-white) the irritant action characteristic of silver is moderated considerably. The *silver-copper* (green), *silver-cadmium* (bright yellow), *silver-mercury* (bright yellow), and *silver-nickel* compounds are also described.

R. BRIGHTMAN.

Preparation of *N*-aralkylated aromatic amines. J. D. RIEDEL A.-G. (G.P. 423,132, 4.3.24).—Arylideneamines (Schiff's bases) are treated with hydrogen or gases containing hydrogen below 180° in the presence of a catalyst formed from one or two metals of the iron group or their oxides or salts, preferably with addition of a heavy metal not belonging to the iron group such as copper, or its oxide or a salt. The C : N linking is thereby smoothly reduced, and an almost quantitative yield of the corresponding *N*-aralkylamine is obtained. For example, *N*-benzal-*p*-phenetidine dissolved in decahydronaphthalene is treated with hydrogen under a pressure of 5 atm. at 90—100° in presence of about 5% of Ni-Co-Cu catalyst to give *N-benzyl*-p-*phenetidine*, m.p. 48°, in quantitative yield. *NN*-Dibenzylidene-*p*-phenylenediamine similarly treated gives *NN-dibenzyl*-p-*phenylenediamine*, m.p. 102°. *N*-Cinnamylidene-*p*-phenetidine treated in hexahydrotoluene with hydrogen under pressure at 100° in presence of a Ni-Cu catalyst yields *Nγ-phenylpropyl*-p-*phenetidine*, m.p. 34°, b.p. 230—235°/14 mm.

T. S. WHEELER.

Preparation of complex silver salts. E. MERCK, Assees. of K. ROTH (G.P. 423,231, 17.6.23).—White or yellowish-white complex silver salts, giving colourless solutions in water, and possessing greater disinfecting powers than silver alkali cyanides, are obtained by dissolving the silver salts of bile acids by means of alkali cyanides and precipitating with alcohol or similar precipitants, or evaporating to dryness on the water-bath or in a vacuum. Thus a suspension of silver cholalate, cholate, glycocholate, taurocholate, choleinate, or desoxycholate may be brought into solution with potassium, sodium, or ammonium cyanide.

R. BRIGHTMAN.

Preparation of rhodinol. FARBENFABR. VORM. F. BAYER & Co., Assees. of L. TAUB, A. WINGLER, and W. SCHULEMANN (G.P. 423,544, 10.1.24).—

Magnesium methylheptenyl halides and ethylene glycol derivatives, such as ethylene oxide or ethylene halogenhydrins are allowed to interact and the product is decomposed in the usual way. Thus methylheptenyl bromide (α-bromo-αε-dimethyl-$\Delta^{\delta\epsilon}$-hexene), b.p. 89°/20 mm., and magnesium in dry ether are treated with ethylene oxide and decomposed with acetic acid and ice-water. The rhodinol obtained is free from by-products and has b.p. 113—115°/14. mm. R. Brightman.

Preparation of acid sodium formate. R. Koepp & Co., and E. Elöd (G.P. 424,017, 27.6.23. Conv., 5.9.22).—Sodium formate is introduced into aqueous formic acid of at least 20% strength at ordinary or raised temperature. To obtain sodium diformate aqueous formic acid of not less than 50% strength is used. By working with a weaker solution trisodium hydrogen formate results, which by treatment with 60% formic acid is converted into sodium diformate. Examples for the production of trisodium hydrogen formate, of sodium diformate, as well as of the conversion of trisodium formate into sodium diformate are given. The acid formates separate, on cooling the mixture to 15°, as well-defined crystals, which can be separated easily from the mother liquor. W. G. Carey.

Preparation of protocatechuic acid. E. Heuser (G.P. 424,542, 16.1.24).—Lignin, lignin derivatives, or lignin-containing waste products are fused with potassium hydroxide in a nickel or iron crucible with addition of ammonium carbonate in an atmosphere of hydrogen. 18—20% of pure protocatechuic acid is obtained. The small quantities of pyrocatechol produced are removed by extraction with benzene. L. M. Clark.

Purification of thionyl-*p*-azo-*o*-aminotoluene. Kalle & Co., Assees. of E. Spöngerts (G.P. 424,613, 15.1.24).—The product obtained by treatment of *p*-azo-*o*-aminotoluene in toluene solution with thionyl chloride at 100° is freed from excess of solvent and reagent, and is repeatedly stirred with vaseline or wool fat at 80°. Filtration from insoluble impurities yields a product containing pure thionyl-*p*-azo-*o*-aminotoluene, which finds direct therapeutic application. L. M. Clark.

Preparation of methylaminoacetopyrocatechol dibenzyl ether. E. Merck, Chem. Fabr., Assees. of A. Dützmann and W. Krauss (G.P. 424,659, 27.9.24).—Methylaminoacetopyrocatechol is treated with benzyl bromide in presence of sodium ethoxide in absolute alcoholic solution. The mixture is heated at 100°, poured into water, and the precipitated dibenzyl ether collected. It serves as a starting point for the synthesis of compounds of the adrenaline type. L. M. Clark.

Germicidal suspensions of silver and mercury salts. P. Saxl and E. Kriwatschek (G.P. 423,666, 21.10.22; Austr. P. 101,025, 6.11.22).—1—5% suspensions of the chlorides are prepared when silver or mercurous nitrate solutions are quickly boiled with sodium chloride and gum arabic solutions and rapidly cooled. These preparations are of therapeutic value. L. M. Clark.

Manufacture of normal butyric acid from butyl aldehyde. C. W. Hancock, Assr. to Commercial Solvents Corp. (U.S.P. 1,580,137, 13.4.26. Appl., 31.8.22).—See Can. P. 249,868; B., 1926, 463.

Catalytic reactions (G.P. 423,542).—See I.

Aromatic aminohydroxy-compounds by electrolysis (E.P. 254,204).—See IV.

Bringing about changes in dielectric carbon compounds (E.P. 254,375).—See XI.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Optical sensitisation. II. Water as sensitiser. [Assimilation of carbon dioxide by plants.] G. Kögel and A. Steigmann (Z. wiss. Phot., 1926, **24**, 171—176; cf. B., 1926, 386).—Observations on leuco-Rhodamine 6G give further support to the view that the hydrogen activated by an exposed dye goes to a large extent to other hydrogen acceptors than the dye itself. The fact that chlorophyll is a good optical sensitiser for silver bromide-collodion indicates that it can activate hydrogen which reacts with the silver bromide. The activated hydrogen must come from the moisture present in the air, *i.e.*, water is a sensitiser. Carbon dioxide assimilation by plants is explained as follows: exposed chlorophyll activates the hydrogen atoms of water present, this hydrogen reacts with carbonic acid to give formaldehyde and hydrogen peroxide, and the hydrogen peroxide decomposes in presence of catalases to give water and oxygen. According to this the relation $CO_2/O_2=1$ is explained. Other considerations are given, indicating that water can act as a sensitiser. W. C.

Nature of optical sensitisation and desensitisation. H. H. Schmidt (Z. wiss. Phot., 1926, **24**, 223—227).—The theory of Kögel and Steigmann (B., 1926, 386) does not agree with modern views of the action of light on silver halides, nor with experimental observations on the velocity of bleaching of sensitising and desensitising dyes. In sensitising with dyes, very stable complexes are formed between the dyes and the silver halide. The displacement of the sensitivity spectrum compared with the absorption spectrum of the dye shows that the process of complex formation is accompanied by the evolution of 2000—3000 cals. On exposure an internal transfer of energy occurs, in the case of acid sensitisers from the dye anions to the silver ions in the lattice, and in the case of basic dyes from the dye cation to the lattice bromine or silver ion. Desensitisation is not due to a greater affinity of the dye for hydrogen; Nile Blue, a good desensitiser, is only slowly bleached, while good sensitisers, such as Pinachrome and Pinaverdol, are readily bleached. All useful acid and basic sensitisers and desensitisers give the Methylene Blue effect of Kögel and Steigmann. Sensitisation of the bleaching

of dyes in water and in gelatin by silver halide is regarded as due to the energy absorbed by the halide on exposure, but not used in changing it photochemically; an analogy is given by the experiments of Cario and Frank (Z. Physik, 1922, **11**, 161) on the dissociation of hydrogen by excited mercury atoms. W. CLARK.

Effect of concentration of sensitiser on speed, and fog corrections. S. E. SHEPPARD (Phot. J., 1926, **66**, 399—409).—With increasing amounts of allyl thiocarbamide added to an emulsion, speed increases to a maximum value and then falls off (cf. B., 1925, 785). Fog values progressively increase, and rapidly so when the maximum is passed. With the emulsion used an increase of speed from about 19 to 3000 H. and D. (fog values were not deducted in measuring speeds) was obtained, the maximum value being found with 0·0146 g. of allylthiocarbamide per 100 g. of silver halide. Inherent fog corrections to the characteristic curve are discussed. Evidence indicates that such fog no longer contributes to the effective density at exposures above a certain value, which varies with the fogging coefficient and probably with the development. The bearing of fog upon available speed is discussed in the light of Luther's theory of the under-exposure portion of the characteristic curve (Trans. Faraday Soc., 1923, **19**, 340). The falling off in speed when the maximum has been passed in sensitising with allylthiocarbamide, is probably due to an increase in the number of nuclei per grain. Such an increase would result in a lowering of the chance of a given nucleus increasing to developable size on a given exposure. W. CLARK.

PATENTS.

Synthetic resins, photographic process and media. M. C. BEEBE, A. MURRAY, and H. V. HERLINGER, Assrs. to WADSWORTH WATCH CASE CO. (U.S.P. 1,587,269, 1.6.26. Appl., 18.11.22).—A photographic coating for application to a metallic surface is composed of a resin derived from the condensation of a five-membered monoheterocyclic compound and a ketone, together with a sensitising agent. The sensitising agent is one that will liberate a halogen. Furfuramide and furfuracetone resins, sensitised with iodoform, for example, are more permanent than asphaltum and are resistant to acids, so that the prints obtained by developing the image formed by the selective action of light can be used for etching in relief and intaglio. E. S. KREIS.

Condensation photographic process and media. M. C. BEEBE, A. MURRAY, and H. V. HERLINGER, Assrs. to WADSWORTH WATCH CASE CO. (U.S.P. 1,587,270, 1.6.26. Appl., 24.11.22).—The condensation product of a phenol and a compound containing an active methylene group, is used in conjunction with a sensitising agent which may be a halogen-substituted hydrocarbon with or without a metal halide. The image formed by the selective action of light may be developed by selective dyeing, the use of various solvents, or the application of soap and water. E. S. KREIS.

Natural resin photographic medium and process. M. C. BEEBE and A. MURRAY, Assrs. to WADSWORTH WATCH CASE CO. (U.S.P. 1,587,271, 1.6.26. Appl., 27.11.22).—The more sensitive fractions of asphaltum, bitumen, or any other light-sensitive and suitable resin are incorporated in a photographic coating with a colloidal halide as sensitiser. The sensitising agent should be one that will not affect the resin in the dark and is soluble in the coating. The specification mentions lead tetraethyl or iodoform in methyl ethyl ketone with a trace of ammonia added. A suitable developer for the above-mentioned resins is a mixture of equal parts of oleic acid and white mineral oil. E. S. KREIS.

Synthetic resins, photographic process and media. M. C. BEEBE, A. MURRAY, and H. V. HERLINGER, Assrs. to WADSWORTH WATCH CASE CO. (U.S.P. [A] 1,587,272 and [B] 1,587,273, 1.6.26. Appl., 19.1.23).—(A) A light-sensitive coating is made from a sensitising agent and a resin capable of being condensed under the action of light, as those formed from benzaldehyde and aniline or dimethylaniline, phenol or aniline and formaldehyde, or by refluxing benzyl chloride in the presence of powdered ferric chloride and sulphuric acid. (B) The resin may be derived from an amine and a five-membered monoheterocyclic compound, and the following resins are described: those resulting from the condensation of furfuraldehyde and an amine such as β-naphthylamine, *m*- or *p*-toluylenediamine or phenylenediamine, benzidine, or xylidine; from furfuraldehyde and *cyclo*hexanone; and from β-naphthylamine and formaldehyde or benzaldehyde. The sensitising agent is a halogen derivative of a hydrocarbon, such as iodoform, capable of liberating halogen, with the addition if necessary of an auxiliary sensitising agent. E. S. KREIS.

Photographic process and media. M. C. BEEBE and A. MURRAY, Assrs. to WADSWORTH WATCH CASE CO. (U.S.P. 1,587,274, 1.6.26. Appl. 22.1.23).—A hydrophobic colloid synthetic resin is combined with a sensitising agent capable of liberating halogen, *e.g.*, a halogen-substituted hydrocarbon used in conjunction with an auxiliary sensitising agent, and an aromatic solvent. The resins may be derived from the unsaturated carbocyclic series or the aliphatic series, and may include esters and glycerides, the essential condition being that they are capable of being transformed by the selective action of light, to yield a print that can be developed. This developing process is purely physical, the developer being one that will dissolve out the unaltered original constituent. E. S. KREIS.

XXII.—EXPLOSIVES; MATCHES.

Direct method for testing initiating explosives (detonators). L. WÖHLER (Z. ges. Schiess- u. Sprengstoffw., 1925, **20**, 145—150, 165—169; 1926, **21**, 1—5, 35—38, 55—57, 97—99, 121—123).—The strength of detonators may be measured quantitatively by testing their initiating effect on 2 g. of

trotyl (trinitrotoluene) or trixyl (trinitroxylene) phlegmatised with a paraffin oil. Directions are given for the preparation of a sample of trotyl of definite crystal size. Equal weights of the trotyl are then phlegmatised with a standard paraffin oil to yield a series of samples with evenly increasing amounts of paraffin oil. The test is carried out by exploding with the detonator under test 2 g. of each grade of trotyl in a copper tube of 10 mm. diam. placed on a plate 5 mm. thick. The maximum percentage of paraffin oil which still allows complete detonation by the detonator is taken as a numerical measure of the strength of the detonator. The occurrence of complete detonation is determined by noting the loss of weight caused by the explosion and plotting the results with loss in weight and paraffin content as co-ordinates. The points will lie on a line which changes its direction when the detonation is just complete. Comparisons have been made of the initiating effects of increasing amounts of trotyl, tetryl, and tetranitropentaerythritol in detonators. Detonators with an indented or perforated base lose in initiating effect on a surrounding explosive owing to their diminsihed lateral effect, although in the ordinary lead plate test they appear to be more powerful than the ordinary type. The increased effect of detonators such as the Briska in which the charge is compressed at 250—2000 kg. per sq. cm. is confirmed. The increase is greater for tetryl and tetranitropentaerythritol than for trotyl. Two commercial types were tested by the method. A No. 8 detonator with 0·9 g. of trotyl and 0·5 g. of crystalline fulminate in a copper tube proved to be weaker than a detonator of the same size with 0·85 g. of tetryl and 0·3 g. of azide–styphnate mixture in an aluminium tube. The higher initiating power of tetryl over trotyl is not observed if fulminate is used. Fulminate and azide show the same results with small amounts of tetryl (0·6 g.), but larger amounts of trotyl, tetryl, and tetranitropentaerythritol are much more effective with even small amounts of azide than with fulminate.

S. Binning.

XXIII.—SANITATION; WATER PURIFICATION.

Works experience in the supervision of rapid filtration plant [for the purification of water]. F. Egger (Z. angew. Chem., 1926, **39**, 962—964).—At the Stuttgart waterworks 30,000 m³. of water per day are treated, most of which is drawn from the River Neckar and is polluted by industrial and domestic effluents. Addition of 20 mg. of aluminium sulphate per litre gave the best results for flocculation of the impurities on the large scale, although preliminary laboratory tests indicated the necyessit of adding 60 mg. per litre. After settling for 2½—4 hrs. rapid filtration gave a filtrate consuming 9·4 mg. of permanganate per litre and slow filtration on a sand filter one consuming 10 mg. per litre, against a consumption of 15·5 mg. for the original water. Rapid filtration reduced the chlorine consumption from 0·3 mg. per litre to 0·13 mg., and the bacteria content of the filtrate was only 10% of that of the original water; no change in the hardness was effected. Tests on a relatively pure and somewhat softer lake water gave similar results except that the high content of carbon dioxide retarded the flocculation of the precipitate. This difficulty was overcome by agitating the water after addition of the aluminium sulphate. The only disadvantage of the rapid filter compared with sand filters appears to be the difficulty of removing completely the bacteria; hence the filtered water must subsequently be treated with chlorine.

A. R. Powell.

Double chlorination [of water]. C. R. Cox (J. Amer. Water Works Assoc., 1926, **16**, 55—71).—Experiences at numerous plants where chlorine is applied to the raw water prior to filtration (prechlorination) and where it is applied to both raw and filtered water (double or "split" chlorination) are reviewed (cf. B., 1922, 994 A; 1924, 441, 613). Double chlorination provides a flexible and economical means of reducing and equalising the bacterial load on filters treating heavily polluted water, improving coagulation, oxidising iron, precipitating sulphur compounds, altering taste-producing substances, and preventing organic growths from developing in coagulation basins and filters, and furnishes in addition reserve treatment in case of temporary failure of the preliminary process.

W. T. Lockett.

Manuring experiments with sewage. Zielstorff, Keller, and Spuhrmann.—See XVI.

Patents.

Purification of industrial effluents containing nitrogen. A. V. A. Girard (F.P. 597,515, 1.5.25. Conv., 10.5.24).—Waste liquor containing nitrogen compounds, such as tannery or slaughter-house effluent is treated with the usual precipitating and coagulating agents and passed slowly through a long tank fitted with baffles, into which fecal matter is discharged and in which fermentation is thus set up. The fermentation and consequent purification is completed by passing the liquor over a bacterial bed.

T. S. Wheeler.

Manufacture of mixtures yielding salts of sulphochloroamides. Farbenfabr. vorm. F. Bayer & Co. (E.P. 241,580, 17.10.25. Conv., 18.10.24).—Organic sulphonamides and their salts form stable mixtures with high-percentage calcium hypochlorite. On adding the mixtures to water salts of sulphochloroamides are instantly formed. The mixtures can be used for all purposes for which hypochlorite and chloroamide are used. A salt may be added to the mixture to precipitate the calcium in such cases as washing and bleaching, where insoluble calcium soaps would otherwise be formed. Typical mixtures are 22·9 pts. of calcium hypochlorite (62·1% of available chlorine) with 34·4 pts. of *p*-toluenesulphonamide; or 22·9 pts. of calcium hypochlorite with 42 pts. of potassium *p*-toluenesulphonamide and 19·5 pts. of sodium sulphate decahydrate.

B. Fullman.

Gases from combustion of sulphur for fumigation (E.P. 230,441).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCTOBER 1, 1926.

I.—GENERAL; PLANT; MACHINERY.

Measurement of currents of air and gases, with especial reference to dynamic principles. O. Mattner (Chem.-Ztg., 1926, **50**, 533—534).—The method of measuring a flow of gas by determining the pressure drop at a constriction in the pipe is quite general, and its application depends only on a suitable choice of the type of orifice and method of pressure measurement. The static gas pressure can be determined by Ser's or Nipher's apparatus (perforated plates parallel to the gas stream). The total pressure is indicated by a Pitot tube; and the dynamic pressure, the difference between the two, can be ascertained by connecting the two devices to a differential manometer. This is the principle of the "pneumometers" of Krell, Prandtl, and Brabbée. The average velocity of a current of gas is given by the formula $v = \sqrt{2g\,Pd/\gamma k}$, where Pd is the pressure difference on the two sides of a perforated plate or similar device, γ is the specific gravity of the gas, and k a constant depending on the apparatus. Types of "pneumometers" are described.

C. Irwin.

Studies in adhesion. I. W. Hardy and M. Nottage (Proc. Roy. Soc., 1926, **A**, **112**, 62—75).—A cylinder standing in a pool of lubricant on a plate can be lifted by any force great enough to overcome the slight resistance offered by the surface tension of the lubricant, provided sufficient time is given. Adhesion is the normal pull required to detach the cylinder instantaneously from the plate. To obtain comparable values, cylinder, plate, and lubricant must be in a mechanically corresponding relation; this occurs when the thickness of the layer of lubricant is such that the Leslie pressure carries the load, and to the force needed to break the cylinder away from this equilibrium position the name "A value" is given. For all loads employed, the layer of lubricant corresponding to this position was found to be of sensible thickness. The latent period which elapses before the steady state is reached is given for a number of lubricants, and is assumed to be due either to the orientation of the lubricant molecules in the attraction fields of the solids, or to the cylinder rising or falling in the pool of lubricant. The A values for 24 lubricants using different solids and different loads are tabulated, and the effects of pressure, of the nature of the solid, of the molecular weight of the lubricant, and of temperature, are discussed. The coefficient α $(= A/\text{load})$ decreases as the load increases, up to the highest load tested (259·6 g.), and the A value is a linear function of the molecular weight of the lubricant, for any one chemical series, and of the temperature. The effect of a change in the nature of the solid is to shift the curve for A and molecular weight parallel to itself, and the curve for two different solids is half-way between the curves for each solid by itself. It is suggested that the influence of the nature of the solid wall should be taken into account in certain of the standard methods of viscosity measurement.

L. L. Bircumshaw.

Validity of flicker photometer measurements in heterochromatic photometry. A. H. Taylor (J. Opt. Soc. Amer., 1926, **13**, 193—204).

See also A., Sept., 898, **Adsorptive powers of charcoal** (Ogawa); **Adsorption by ash-free adsorbent charcoals. Purification of adsorbent charcoals** (Miller). 899, **Adsorption of dissolved substances** (Charriou).

Percolator. Rattray.—See XX.

Patents.

Method of grinding or crushing. E. Barthelmess (E.P. 251,665, 3.5.26. Conv., 2.5.25).—The material is subjected to centrifugal impact by means of a fluid entering the grinding chamber tangentially at a high speed. From the grinding chamber the mixture is led to a separator and only the fluid returns to the pump or fan, where it receives the energy to effect the grinding and then re-enters the grinding chamber.

B. M. Venables.

Tube mills for grinding or crushing. Wicking'sche Portland-Cement & Wasserkalkwerke, and A. Andreas (E.P. 254,909, 14.9.25. Addn. to 241,174).—Modifications are made in the screening device of a compound ball mill such as is described in the original patent (cf. B., 1926, 344) to improve the transfer of the sifted material to the secondary grinding compartment. To this end the screens may be inclined to the axis of the mill, and may be tangential rather than radial. Curved guides may also be used. Means are also provided for easily changing the screens.

B. M. Venables.

Gyratory cone crusher. E. B. Symons, Assr. to Symons Bros. Co. (U.S.P. 1,592,313, 13.7.26. Appl., 4.6.25).—In a crusher in which a gyrating cone is surrounded by an inverted bowl or the stationary part of the crusher, the latter is mounted resiliently in the frame in such a way that it is always pressed downwards towards the gyrating cone.

B. M. Venables.

Centrifugal machines. V. Redlich (E.P. 252,339, 23.4.26. Conv., 19.5.25).*—A centrifugal machine with

continuous discharge of both solid and liquid separated products comprises an outer basket, 1, 2, and inner feed devices, 19, 22, rotating at the same high speed, also an inner cellular basket, 3, rotating very slightly faster than the above-mentioned rotating parts. The outer basket comprises an inner perforated wall, 1, which forms the filter surface, and an outer non-perforated wall, 2, which serves to collect the clear liquor.

FIG.1.

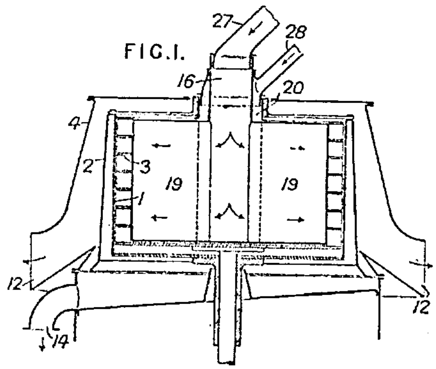
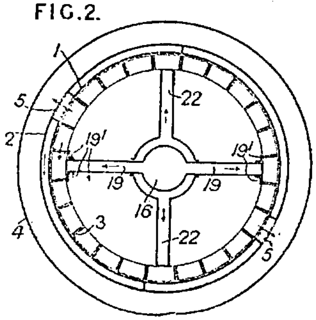


FIG.2.



At intervals slots, 5, are formed through both walls, through which the dried solid matter is discharged, being collected by the fixed outer wall, 4, of the apparatus. The feed of the original material is through passages, 19, to cells that have just left the discharge ports, 5. Passages, 22, serve for the admission of wash liquor at a later stage of the draining. The annular space of the outer basket is divided into segments by partitions, so that the original filtrate and washings may be collected separately. When it is desired to collect the solid matter in pieces of a definite size and shape, *e.g.*, sugar cubes, the cells are made to suit the desired form, and the collecting wall, 4, is made up of a series of guides designed to collect the solid and reduce its speed without sho k.
B. M. VENABLES.

Concentrating solutions, even those liable to incrust to a great extent and corrosive. P. BRINGHENTI (E.P. 254,944, 7.11.25).—The apparatus comprises a preheating vessel where an inert liquid, *e.g.*, melted paraffin, is heated by any suitable external source, a heating vessel in which the solution to be evaporated is heated by contact with sprays or streams of the inert liquid, the latter afterwards rising to the top, and a separator in which the inert liquid is freed from any entrained solution, the latter returning to the heating tank and the former to the preheater. The heated solution falls or is pumped to a second vessel, where it is permitted to evaporate, the residual solution being returned to the heating vessel and the vapour being condensed preferably in the preheater of another similar effect operating at a lower pressure. B. M. VENABLES.

Filter-medium support. S. E. WOODWORTH, Assr. to HAMILTON, BEAUCHAMP, & WOODWORTH (U.S.P. 1,593,051, 20.7.26. Appl., 30.1.24).—Spaced strips, set at an angle to the longitudinal axis of the filter drum, are placed between the drum and the filter medium surrounding it. H. HOLMES.

Removal of vapours or gases from moist gaseous mixtures. BADISCHE ANILIN- & SODA-FABR. (F.P. 604,207, 3.7.25. Conv., 5.9.24).—An absorbent, such as silica gel and, if necessary, activated charcoal, is used in several vessels, through which the gas passes in succession.
W. G. CAREY.

Process of drying materials. FULLER FUEL CO., Assees. of M. D. JONES (E.P. 231,151, 3.3.25. Conv., 18.3.24).—See U.S.P. 1,568,738; B., 1926, 349.

Refrigerating systems. STITT REFRIGERATION CO., Assees. of R. R. STITT (E.P. 246,814, 6.1.26. Conv., 2.2.25).—See U.S.P. 1,570,080; B., 1926, 224.

Purifying, clarifying, decolorising and deodorising liquids or gases. H. W. A. BRANCO (E.P. 256,316, 4.5.25).—See F.P. 598,826; B., 1926, 304.

Refrigerating machine. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of C. ORR (E.P. 244,768, 12.12.25. Conv., 16.12.24).

Self-acting apparatus for mechanical separation of liquid mixtures. H. HITCHON (E.P. 256,012, 19.6.25).

Protection of metallic surfaces against incrustation. A. L. MOND. From W. THALHOFER (E.P. 256,481, 27.1.26).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Reduction of carbon monoxide. H. TROPSCH, A. SCHELLENBERG, and A. VON PHILIPPOVICH (Abhandl. Kennt. Kohle, 1925, 7, 63—67; Chem. Zentr., 1926, II, 847—848).—The formation of methane from a mixture of carbon monoxide (1 vol.) and hydrogen (3 vols.) in contact with nickel, which normally occurs quantitatively at 240—250°, is greatly diminished by adding ammonia to the gas mixture. Neither formaldehyde nor hexamethylenetetramine was formed, and the final mixture contained 13% of methane, 15% of carbon monoxide, 64% of hydrogen, and 8% of nitrogen. With a nickel catalyst which had been treated with alkali, the same gas mixture yielded, at 400° and atmospheric pressure, 53% of methane and 1·1% of unsaturated hydrocarbons, the only liquid reaction product being water. The same reaction occurred with a nickel

catalyst which had not been treated with alkali, except that no unsaturated hydrocarbons were formed. A mixture of equal volumes of carbon monoxide and hydrogen, in contact with an alkaline nickel catalyst at 400°, deposited much carbon. With a feebly alkaline catalyst the final gas contained 27% of carbon dioxide, 2% of carbon monoxide, 26% of hydrogen, and 45% of methane, whilst with a more strongly alkaline catalyst it contained 11% of carbon dioxide, 29% of carbon monoxide, 50% of hydrogen, and 9% of methane. With both alkaline and neutral catalysts the methane contained 1·5% of higher homologues, reckoned as ethane. A mixture of equal volumes of carbon dioxide and hydrogen, in contact with neutral nickel at 400°, gave a final gas containing 10% of carbon dioxide, 42% of hydrogen, and 48% of methane, whilst with alkaline nickel it gave 7% of carbon dioxide, 70% of hydrogen, and 23% of methane. In no case could methyl alcohol be detected.

W. T. K. BRAUNHOLTZ.

Reduction of carbon monoxide by hydrogen in contact with a heated iron spiral in a hot-cold tube. F. FISCHER and A. JAEGER (Abhandl. Kennt. Kohle, 1925, 7, 68—74; Chem. Zentr., 1926, II, 847).—When an electrically-heated iron wire spiral was brought to red heat in a mixture of 15% of carbon dioxide, 23% of carbon monoxide, and 56% of hydrogen, contained at 120—150 atm. in an iron autoclave surrounded by cold water, the pressure fell to 70—80 atm. and a gas mixture containing about 9% of carbon dioxide, 40% of carbon monoxide, 22—29% of hydrogen, and 15—19% of methane hydrocarbons was formed. Water containing a considerable amount of formaldehyde was also produced, and solid carbon was deposited on the spiral. By heating the spiral more carefully (to about 620°) the hydrogen disappeared almost completely and a larger amount of saturated hydrocarbons was produced, together with much free carbon and an aqueous liquid of acid reaction containing formaldehyde. The results were practically identical if the iron spiral was previously treated with alkali, except that the yield of formaldehyde appeared to be less.

W. T. K. BRAUNHOLTZ.

Determination of traces of water in mineral oils. W. BOLLER (Chem.-Ztg., 1926, 50, 537—538).—The water vapour evolved on heating the oil is passed through a calcium carbide tube and the acetylene liberated determined as copper acetylide. The carbide tube, 50 cm. long, is arranged within an iron tube and mounted in an air oven. After a preliminary drying of the carbide until free from acetylene, pure and dry hydrogen is passed through the apparatus and the oil sample heated to 140° in an oil bath. After 1 hr. the carbide tube is heated to 180—200° and maintained at this temperature for 1 hr. The absorbent consists of 10 c.c. of 1 : 10 copper sulphate solution to which 4 c.c. of concentrated ammonia and then 3 g. of hydroxylamine hydrochloride have been added, and the whole diluted with water to 30 c.c. The precipitated copper acetylide, after washing with 2% ammonia solution, is converted into cupric oxide or determined volumetrically by its reaction with ferric sulphate. Results as found in the case of an oil containing 0·01% of water are good, though tending to be slightly low.

C. IRWIN.

Improvement of the lubricating efficiency of oils by graphite, and its study by the aid of measurements of the heat of wetting. W. BACHMANN and C. BRIEGER (Kolloid-Z., 1926, 39, 334—346).—The parellelism between lubricating efficiency and heat of wetting previously demonstrated (B., 1925, 435) is applied to explain the improved efficiency of oils to which graphite, especially colloidal graphite, has been added. Measurements show that the wetting heat oil/graphite is with a good oil 7—10 times greater than that of oil/metal. The graphite forms a layer on the surface of the metal so that the efficiency of the lubricant is determined by the wetting heat oil/graphite. Almost all the oils investigated (good and bad) show the same wetting heat against graphite so that they are all raised to the same efficiency by its addition. The improvement brought about by the addition of a small percentage of an unsaturated fatty acid is also explained by the elevation of the heat of wetting which it causes.

N. H. HARTSHORNE.

See also A., Sept., 893, **Determination of heats of combustion of volatile substances in the calorimetric bomb** (VERKADE and COOPS). 909, **Isothermal calorimetry** (VON WARTENBERG and LERNER-STEINBERG).

Ethylene obtained by cracking of petroleum for production of alcohol. GERR and POPOV.—See XX.

PATENTS.

Transformation of lignites, coals, and other carbonaceous matter into fuels of high calorific power, including smokeless fuels. H. DEBAUCHE (E.P. 254,964, 28.11.25).—The carbonaceous matter is first subjected to low-temperature distillation, and the hot semi-coke is screened, graded, mixed, and agglomerated with a suitable binder (*e.g.*, pitch), all the operations being carried on continuously. W. T. K. BRAUNHOLTZ.

Treatment of peat. E. H. WINTER (E.P. 254,994, 16.2.26).—A hard and dense fuel is obtained from peat, without the necessity of expensive drying operations, by mixing the peat with a caustic substance (*e.g.*, lime), an amylose carbohydrate (*e.g.*, starch), an alum, a sugar (*e.g.*, glucose, molasses), and an oxidiser or nitrate, and allowing the mixture to dry in the air. The materials are added, in aqueous solution or emulsion, while the peat is being macerated or disintegrated, a suitable mixture being 8000 pts. of wet peat, 40 pts. of lime, 10 pts. of starch, 1 pt. of alum, 2 pts. of saltpetre, and 12 pts. of maize-sugar or confectioner's glucose. A heat-insulating or packing material can be made in like manner, the nitrate being omitted and the quantity of the other added materials being reduced.

W. T. K. BRAUNHOLTZ.

Device for measuring flue-gas losses. T. STEIN, Assr. to GENERAL ELECTRIC CO. (U.S.P. 1,591,444,

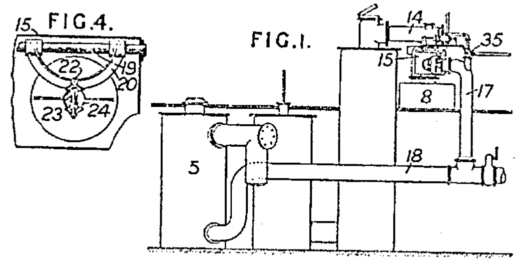
6.7.26. Appl., 10.2.25. Conv., 28.2.24).—The apparatus is designed to operate in accordance with the following modification of the Siegert formula: $V = (W + c_1/k_n)(\Delta t + c_2k_2)$, where V = total flue gas loss, W = content of unburned material in flue gas, k_2 = water content of flue gas, $k_n = CO_2$ content of flue gas after re-burning, Δt = temperature difference between flue gas and entering air; c_1 and c_2 are constants. Three devices are provided for measuring, respectively, W, k_n, and Δt, and these are connected to a measuring device which indicates the flue gas loss in any suitable units.
R. B. CLARKE.

Gas generating plants. A. BREISIG (E.P. 235,891, 17.6.25. Conv., 18.6.24).—To obviate the use of a large relief holder for water-gas plants a heavy gas holder, with a capacity of only $\frac{1}{20}$ to $\frac{1}{100}$ of the hourly output and of sufficient weight to drive the gas through the purifying apparatus is installed between the generator and condensers. A throttle valve is fitted between the gas holder and the condensers, and is so adjusted that rather more than the average hourly output will pass through the valve and the holder will come right down at the end of each blowing period.
A. C. MONKHOUSE.

Manufacture of a mixture of water-gas and coal-gas. A. BREISIG (E.P. 241,902, 20.10.25. Conv., 21.10.24. Addn. to 207,651).—The method of carbonising fuels by internal heating and the use of heat accumulators as described in the chief patent (B., 1924, 122), is modified so that in the preliminary carbonisation of the coal, coke in excess of that required for the producer is made, thus producing a richer gas; the extra heat for carbonisation is provided by increasing the blow period.
A. C. MONKHOUSE.

Air-blast device for semi-water-gas producers. SOC. ANON. D'EXPLOIT. DES BREVETS "COUSIN" DITE "LE CHAUFFAGE IND." (E.P. 242,597, 14.8.25. Conv., 6.11.24).—A water tank is arranged centrally at the base of the producer, and an air blast is delivered to the centre of the latter under a bell, the lower edge of which extends below the water level in the tank, so that the air blast carries forward a fine spray of water which will instantly be vaporised. A cover is supported above the water tank so as to distribute the moist air blast below the zone of clinker formation, or it is arranged in a stepped disposition to afford the blast exits at various heights. The level of the water in the tank can be adjusted or can be maintained constant by means of an overflow. The air-blast device may be used in a producer with water-sealed ash-pit, the water tank being constituted by the ash-pit itself or by a tank immersed in it.
W. T. K. BRAUNHOLTZ.

Water-gas manufacture. G. R. STEERE and J. B. EBERLEIN (E.P. 254,064, 19.5.25).*—A "back-run" water-gas plant is provided with a swinging valve which allows the wash-box to be put into communication with either the superheater or the back-run pipe. The valve is operated by the ram of a hydraulic cylinder, which also actuates a two-way steam cock in such a way that steam enters the generator either via the "back-run" pipe or via the superheater and the carburettor, according to the position of the valve. During the "blow" the swinging valve (see Fig. 4) is in the position indicated in Fig. 1,



and the air blast enters the generator via 18. During an "up-run" steam enters the bottom of the generator via 17 and 18. When using the "back-run" the swinging valve is reversed, so as to cut off 14 from the wash-box 8, and steam is admitted through 14 to the superheater, whence it passes through the carburettor to the top of the generator. The gas made during the "back-run" leaves the generator through 18 and enters the wash-box via 17. Thus 18 serves as blast main, a "back-run" pipe, and a steam main to the generator.
S. PEXTON.

Pressure-gas producers. K. KOLLER (E.P. 254,392, 2.4.25).—To enable the steam–air mixture to be correctly distributed at the base of a grateless gas producer without obstructing the passage of ash and clinker, a distributor in the form of a short cylindrical pipe or dome in which are openings is used. The outlet openings are regulated by slides in the blast nozzle, thus altering the velocity of the emerging mixture; the steam and air are supplied through channels in the distributor in order to keep it cool. Another form of distributor consists of rings separated by the nozzle openings with a common space in the cap or dome; the path of the steam–air mixture in this case is reversed before emerging at the nozzles and adjustment is made by a rotary slide.
A. C. MONKHOUSE.

Construction of regenerative coke-oven flues. R. F. F. FABRY (E.P. 254,652, 24.3.26).—The principles of E.P. 105,047 (B., 1917, 497) are applied to regenerative ovens with vertical flues. Each vertical flue has a nozzle at its lower end which serves alternately as air inlet and as outlet for the products of combustion; a single gas inlet common to both vertical flues is arranged under the partition which divides them in order to direct the supply of combustible gas always into that vertical flue which for the time receives an ascending supply of hot air from the corresponding regenerator. The gases are diluted by the products of combustion descending the other vertical flue.
A. C. MONKHOUSE.

Coke ovens. H. SCHRÖDER (E.P. 254,960, 24.11.25).—In horizontal coke ovens, having regenerators beneath and parallel to the oven chambers, the substructure supporting the chambers and heating walls is strengthened by the provision of arches extending over the width of a number of ovens and each containing a regenerator separated from the adjacent ones by supporting walls.

Each regenerator is covered by two arches, one for support and the other permitting expansion of the brickwork. W. T. K. BRAUNHOLTZ.

Manufacture of coke. H. J. TOOGOOD (E.P. 254,811, 14.4.25).—Metallic retorts are divided by longitudinal and transverse partitions into narrow compartments, open at the top and bottom, and a machine travels along the gasworks retort house and inserts and removes these retorts bodily into and from other retorts, such as the usual gasworks retorts. The stoking machines are provided with means for quenching the discharged coke and utilising its heat for drying and preheating coal for future carbonisation, and also with means for grading the fuel and compacting it as it is fed into the metallic retorts. W. T. K. BRAUNHOLTZ.

Coking coal. M. W. DITTO and W. M. DUNCAN (U.S.P. 1,591,023, 6.7.26. Appl., 29.3.23).—A retort, divided by a vertical wall into two chambers, is fed by a conveyor below the retort. The two chambers are sealed by a rolling contact between the division wall and the coal in the conveyor. Fuel is burnt with air in the one chamber and the products of combustion carbonise the fuel in the other chamber. A. C. MONKHOUSE.

Distilling bituminous coal with recovery of gaseous distillate. R. M. FOLSOM and C. A. RAYMOND, Assrs. to NEW ENGLAND FUEL & TRANSPORTATION Co. (U.S.P. 1,591,672, 6.7.26. Appl., 19.8.25).—Coal is carbonised under suction in a battery of coke ovens with overhead charging. To prevent loss of gaseous products the suction is increased during charging and the products are withdrawn at the opposite sides of the charging openings. A. C. MONKHOUSE.

Making activated carbon. H. RODMAN, Assr. to RODMAN CHEMICAL Co. (U.S.P. 1,591,235, 6.7.26. Appl., 21.8.18).—Carbon which is kept in motion is heated in a furnace to the required temperature by direct contact with hot gases. A. C. MONKHOUSE.

Absorbent charcoal. E. URBAIN (F.P. 604,181, 31.12.24).—Activated charcoal is made under such conditions that products of different density are obtained which exhibit different activity towards the same gases and vapours. W. G. CAREY.

Activated charcoal. J. MAGTEGAAL (F.P. 604,417, 10.10.25. Conv., 15.10.24).—The gases from the dry distillation of carbonaceous material, cooled and purified if necessary, are led from the retorts into a furnace, where they are burned and pass over incandescent carbon. W. G. CAREY.

Preparation of a highly active charcoal. I. DEIGLMAYR CHEM. FABR. A.-G., Assees. of H. SCHWARF (G.P. 430,031, 24.5.23).—A mixture of beet molasses and brewers' grains is carbonised in presence of potassium carbonate, the product is washed with dilute acid, and ignited again in the absence of air. W. G. CAREY.

Filling mass for containers for explosive gases. C. NESS, Assr. to PREST-O-LITE Co. (U.S.P. 1,591,397, 6.7.26. Appl., 24.8.25).—A mixture of plaster of Paris, water, and acetone is used as a filling mass for receptacles for storing explosive gases. A. C. MONKHOUSE.

Treating motor fuels. T. MIDGLEY, JUN., Assr. to GEN. MOTORS CORP. (U.S.P. 1,592,953, 20.7.26. Appl., 4.10.22).—A solid pellet for adding to motor fuels contains lead tetraethyl and *p*-toluidine. H. MOORE.

[Motor] fuel. T. MIDGLEY, JUN., Assr. to GEN. MOTORS CORP. (U.S.P. 1,592,954, 20.7.26. Appl., 19.5.23).—A motor fuel is mixed to a homogeneous liquid with an anti-knock substance containing lead and a substance which combines with the lead during combustion to form a compound having a lower fluxing action than lead oxide. H. MOORE.

Motor fuel. T. MIDGLEY, JUN. (U.S.P. 1,592,955, 20.7.26. Appl., 22.1.26).—An internal-combustion engine fuel contains a volatile phenyl compound of a metal. H. MOORE.

Improving motor fuel. D. R. STEVENS, S. P. MARLEY, and W. A. GRUSE, Assrs. to GULF REFINING Co. (U.S.P. 1,593,040, 20.7.26. Appl., 28.12.25).—A material containing an aluminium halide is treated with a combustible organic liquid, which is then added to motor fuel to improve its knocking qualities. H. MOORE.

Conversion of crude mineral or shale oils or tar oils into light oil or spirit and preparation of light oil or spirit from coal, lignite, or other carbonaceous material. E. SCHULTZ (E.P. 254,011, 25.3.25).—Carbonaceous material is heated in a still to 400°, or to a temperature sufficient to release all volatile matter, and the vapours are led to a converter containing chemicals such as common salt, zinc chloride or oxide, lime, zinc shavings, bauxite, aluminium chloride, or magnesia, and maintained at a temperature up to 350°, but always lower than that of the retort. A retort such as is described in E.P. 252,422 (B., 1926, 654) may be used. Alternatively, crude oil may be heated in a still and the vapours passed to such a converter. The matter in the converter may be supported on a rotating screw. The vapours from the converter are passed to a condenser. The condensate is washed with acid, neutralised, and fractionally distilled. H. MOORE.

Cracking and hydrogenation of hydrocarbon oils. G. W. WALLACE (E.P. 255,159, 17.4.25).—The process is carried out in a horizontal or inclined chamber through which pieces of material, such as carbon, carbon briquettes, firebrick, alumina, etc., are continuously passed, and are agitated by rotary crushers to expose fresh surfaces. The material may be coated with a catalyst such as nickel. The oil is introduced as spray or vapour, and passes in counter-current to the catalytic material, which is circulated by a plunger and drops into a vertical extension of the chamber, from which it may

be withdrawn, and is then returned to the horizontal chamber either immediately or after regenerative treatment in the hot state with gas, air, or steam. Air may be introduced with a portion of the oil to form a combustion zone either within or without the chamber. The oil before entering the chamber may be preheated by the combustion of the gases produced by cracking. Hydrogen or steam, or producer gas generated by the action of air and steam on the hot carbon deposited on the material, may be introduced to assist hydrogenation. The cracked vapours are withdrawn by suction near the end where the contact material is introduced.
H. MOORE.

Refining oil. B. V. STOLL (U.S.P. 1,593,275, 20.7.26. Appl., 26.10.22).—Oil is cracked by heating in a coil to about 370—400° under a pressure of about 150 lb. per sq. in. and the product expanded in a cylinder still at about 200—260° under a pressure of about 100 lb. per sq. in.
H. MOORE.

Treating hydrocarbon gases. J. E. KOBERNIK, Assr. to NEWTON PROCESS MANUF. Co. (U.S.P. 1,594,014, 27.7.26. Appl., 27.10.25).—A hot absorbing medium containing hydrocarbons is brought into intimate contact with a hot lean hydrocarbon gas, and the gas and the medium are cooled and subsequently again brought into intimate contact, these operations being repeated as often as is necessary. The medium is afterwards distilled to recover a commercial stabilised gasoline.
H. MOORE.

Apparatus for distilling oils. L. E. LEE (U.S.P. 1,594,296, 27.7.26. Appl., 9.11.21).—The still has a vertical inner shell, against the upper end of which a nozzle projects a jet of oil, and from which the oil is projected on to a dome above the upper end of the shell. The oil is atomised by its impact against the dome, and thence directed to the outer surface of the inner shell, which is heated to vaporise the oil passing over it. An outer shell surrounding the inner shell forms a space to receive the vapours.
H. MOORE.

Refining mineral lubricating oils. J. W. WEIR (U.S.P. 1,592,058, 13.7.26. Appl., 14.5.25).—Lubricating oil stock of relatively low viscosity is treated with sulphuric acid until the action is complete, then, without removing the sludge, is mixed with lubricating oil stock of relatively high viscosity, which is acted upon by the acid sludge.
D. WOODROFFE.

Production of fuel mixtures. T. H. BUTLER, H. W. ROBINSON, and D. W. PARKES (E.P. 256,107, 8.12.25).—See U.S.P. 1,583,573; B., 1926, 572.

Treatment of certain fuels [lignites] to improve their calorific value. W. A. BONE (U.S.P. 1,594,994, 3.8.26. Appl., 5.7.19. Conv., 30.7.18).—See E.P. 130,445; B., 1919, 709 A.

Separation of dry materials [coal]. K. C. APPLEYARD, P. W. BEWICK, J. F. LAYCOCK, M. R. PORTAL, and W. E. MANNERS (E.P. 255,924, 24.4.25).

Coal pulverising machines. A. HERBERT and R. JACKSON (E.P. 255,957, 2.5.25, and 255,966, 5.5.25).

Coke cooling plant. C. SCHWARTZ (E.P. 252,118, 22.12.25. Conv., 12.5.25).

[Quenching] coke when discharged from retorts and the like. DRAKES LTD., and J. W. DRAKE (E.P. 256,142, 29.5.25).

III.—TAR AND TAR PRODUCTS.

Application of the Bergius process to coal tar. L. RHEINFELDER (Mitt. Schles. Kohlenforschungsinst. Kaiser-Wilhelm-Ges., 1925, 2, 34—67; Chem. Zentr., 1926, II., 519—520).—"Berginised" tar yielded light oils which, after removal of acidic and basic constituents, were divisible into three fractions boiling between 70° and 170°, in which benzene, toluene, and xylene respectively could be identified. The neutral oils boiling below 70° appeared to be a mixture of aliphatic or cyclic saturated hydrocarbons with small quantities of unsaturated and aromatic hydrocarbons. Low-boiling aliphatic or cyclic saturated hydrocarbons (perhaps also *cyclo*hexane) were isolated in very small quantities. "Berginisation" of tar has the effect of increasing the content of low-boiling phenols and bases at the expense of the higher homologues. Aniline was isolated in one experiment. De-alkylation appears to take place, and a large quantity of methane hydrocarbons is found in the gases finally leaving the autoclave. The nature of the final products, *i.e.*, the extent of de-alkylation and decomposition, will depend on the temperatures and pressures employed.
W. T. K. BRAUNHOLTZ.

PATENTS.

Conversion of tar oils into light oils (E.P. 254,011).—See II.

Extraction process (G.P. 430,087).—See XX.

IV.—DYESTUFFS AND INTERMEDIATES.

Viscosity and hydration of dye solutions. LIEPATOV.—See A., Sept., 903.

Organic dyes in vulcanised rubber. DRAKELEY.—See XIV.

PATENTS.

Manufacture of condensation products and dyestuffs of the benzanthrone series [*iso*dibenzanthrones]. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABRIK (E.P. 255,277, 7.10.25).—*iso*Dibenzanthrones are obtained by the action of alkaline condensing agents on a mixture of a benzanthrone, not substituted in the 3- and 4-positions, with a Bz-halogenbenzanthrone having a free 4-position. The condensing agents used may be mixtures of caustic alkalis with alkali alkoxides in the presence of an inert diluent, or metal arylides, such as sodium anilide, and oxygen should be excluded during the reaction. By moderating the reaction (*e.g.*, by the

use of a lower temperature), dibenzanthronyls are formed as intermediate products, and these may be further converted into *iso*dibenzanthrones by the action of alkaline or acid condensing agents. For example, a mixture of 20 pts. of benzanthrone and 23 pts. of Bz-chlorobenzanthrone is added, in a current of nitrogen, at 5° to sodium anilide (prepared from 6 pts. of sodium and 400 pts. of aniline), and the mixture stirred for several hours, the temperature being allowed to rise to 12°. After removal of aniline and extraction of the residue with acetone, 3 : 4′-*dibenzanthronyl*, yellow needles, m.p. 326°, is obtained. To convert this into *iso*dibenzanthrone, 10 pts. of it are added, in a current of nitrogen, to sodium anilide (from 3 pts. of sodium and 200 pts. of aniline) at 125° and the mixture is heated at 140—150°. The following additional new compounds are described: 8-*chloro*-3: 3′-*dibenzanthronyl*, brownish-yellow, m.p. above 360°; 9-*chloro*-3 : 4′-*dibenzanthronyl*, m.p. above 360°; 8-*methyl*-3 : 4′-*dibenzanthronyl*, yellowish-green, m.p.above 360°. 8-Chloro*iso*dibenzanthrone dyes cotton in much bluer and stronger shades than *iso*dibenzanthrone.

A. Davidson.

Preparation of sulphurised condensation products [dyes]. I. G. Farbenind. A.-G., Assees. of J. Binapfl and L. Strohmenger (G.P. 427,970, 2.11.23).—Mixtures containing azobenzene or its homologues and aromatic amines or phenols, or the condensation products obtained from azo-compounds and aromatic amines or phenols, are heated with sulphur dichloride or sulphur in the presence of a condensing agent, the coloured products being dyes or capable of conversion into dyes by further heating with concentrated or fuming sulphuric acid. For example, a bluish-green vat dye is obtained by heating at 210° a mixture of azobenzene, *o*-cresol, sulphur, and zinc chloride. A. J. Hall.

Manufacture of [stable] preparations containing diazotised *p*-nitroaniline. I. G. Farbenind. A.-G., Assees. of K. Schnitzspahn (G.P. 426,033, 28.12.24).—Solid sodium nitrite isincorporated in a mixture containing 1 mol. of an aromatic nitroamine having no sulpho- or carboxyl-groups, more than 1 mol. of sulphuric acid (the mixture may also consist partly of equivalent proportions of the sulphate of a nitroamine and bisulphates), and a suitable diluent such as partly dehydrated aluminium sulphate or alum, arylsulphonates or mixtures of arylsulphonates and partly dehydrated alum; the products are very stable and liberate the diazo-compound when dissolved in cold water. A. J. Hall.

Monoazo dyes. W. Duisberg, W. Hentrich, and L. Zeh, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,595,178, 10.8.26. Appl., 26.11.24. Conv., 5.12.23).—See E.P. 225,862; B., 1925, 583.

Halogenated vat dyestuffs of the anthraquinone series. I. G. Farbenind. A.-G., Assees. of A. Holl (U.S.P. 1,595,549, 10.8.26. Appl., 9.6.25. Conv., 20.6.24).—See E.P. 235,919; B., 1925, 703.

Extraction process (G.P. 430,087).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Viscose. II. Ripening of viscose. M. Numa (Cellulose Ind., Tokyo, 1926, **2**, 136—148).—The effects of temperature, light, duration of ripening, and other influences on the ripening of alkali-cellulose have been determined by studying the colloidal properties of cuprammonium hydroxide solutions of the regenerated cellulose, the colloidal properties of the viscose solutions, and the physical properties of the resulting viscose pellicles. The relationship between intensity of colour as measured by a Duboscq colorimeter and the degree of dispersion, observed by Svedberg in the examination of colloidal solutions of gold, has been found to apply in the case of cuprammonium hydroxide solutions of cellulose. The degree of dispersion of alkali-cellulose reaches a maximum after a relatively short period of ripening, and then slowly decreases; this point of maximum dispersion marks the optimum time of ripening, as is confirmed by an examination of the viscose solution and of the films prepared therefrom. Light and temperatures above 20° have a deleterious effect on alkali-cellulose; there is, however, no necessity to conduct the ripening process in the presence of inert gases provided that free access of air is avoided. The best conditions of ripening are, therefore, 5—20 hrs. at a low temperature in a closed, opaque vessel containing as little air as possible.

D. J. Norman.

Esparto pulp by the "Keebra" process. J. d'A. Clark (Pulp and Paper Mag., 1926, **24**, 815).—The "Keebra" process (digestion with pure normal sodium sulphite solution at, for example, 130 lb. per sq. in. for wood and 50 lb. per sq. in. for esparto) offers difficulties in the recovery of the soda: if, however, a modified ("Semi-Keebra") process is adopted, recovery of the soda becomes a practical proposition. This modified process is a soda process in which about 25% of the caustic soda is replaced by its equivalent of normal sodium sulphite; somewhat lower pressures may be used and the yield, though not so high as with the full "Keebra" process, is about 10% higher than that given by the ordinary soda process. "Semi-Keebra" esparto pulp, moreover, gives a stronger sheet of paper with more rattle. "Semi-Keebra" wood pulp resembles soda pulp but is slightly harder, stronger, and more easily bleached.

D. J. Norman.

See also A., Sept., 899, **Adsorbent properties of cellulose nitrate** (Duclaux). 902, **Colloidal characters of cellulose** (Herzog); **Viscose solutions** (Herzog, Gaebel, and Jancke). 903, **Diffusion researches on solutions of cellulose in copper-ammonia solution** (Herzog and Krüger). 935, **Detection of hydroxyl groups of different types. Application to lignin** (Freudenberg and Hess).

Flax wax. Honneyman.—See XII.

Fluorescence of sulphite-cellulose extract. Meunier and Jamet.—See XV.

Nitrous esters of cellulose. Blechta.—See XXII.

Patents.

Treating fibres. Oberrheinische Handelsges. m.b.H., and L. Ubbelohde (E.P. 254,357, 2.3.25).—The process previously described (cf. E.P. 239,605; B., 1925, 877) is modified in that a proportion of the roughening agents is left on the fibre, retention being assisted where necessary by the use of adhesive agents such as grease: alternatively, the fibres may be mixed with an aqueous suspension of the roughening material, centrifuged, dried under pressure, and finally freed from dust. Suitable roughening agents are carborundum, keiselguhr, lime, and the like, but, for long fibres such as artificial silk, materials with softer edges, for example, silica gel, may be satisfactorily used. The retention of roughening agents by the fibre tends to make the yarn more voluminous and more resistant to tearing.

D. J. Norman.

Felting process for woollen and like textile fabrics. E. Ricalens (E.P. 254,609, 19.1.26).—The fabric, after a slight fulling operation, is circulated in a bath containing soap and wool dust until the required quantity of wool dust has been taken up. The treated fabric is then pressed, and washed to remove the impregnating liquor. The soap solution is preferably prepared by saponifying olein with sodium carbonate.

D. J. Norman.

Oxidation (chlorination) of textile materials made from animal fibres, *e.g.*, wool and silk. W. H. Schweitzer (G.P. 430,109, 21.5.24).—Animal fibres, with or without previous treatment with an oxidising agent such as sodium hypochlorite, are treated at high or low temperatures with a solution containing 1% (on the weight of fibre) of sodium *p*-toluenesulphochloramide.

A. J. Hall.

Treatment of artificial [viscose] silk and th like. Viskose A.-G., Assees. of C. Becker and A. Bernstein (E.P. 242,993, 11.11.25. Conv., 14.11.24).—Viscose threads or filaments, coagulated and wound in the usual way, are unwound, while still wet in the acid condition or after washing, and passed successively through an alkaline desulphurising bath and a weak acid bath. The treated threads are finally re-wound in a washing bath.

D. J. Norman.

Improving artificial fibrous materials. L. Lilienfeld (E.P. 253,853, 16.7.25. Conv., 17.6.25).—Cellulose silk yarns in skeins or fabric are treated with caustic soda of concentration not exceeding 5% and then dried before or after removal of the alkali by washing, or souring and washing with water or solutions of inorganic salts (*e.g.*, sodium chloride and ammonium sulphate), the yarns being stretched during at least a part of the treatment; the resulting yarns have an increased strength of 30—100%. For example, viscose, cuprammonium, or Chardonnet silk is simultaneously impregnated with a 0·2—0·3% solution of caustic soda and stretched, then dried, soured with 10% sulphuric acid, washed with water, and dried. Cellulose silk thus treated may be subsequently steamed or heated, and especially good results are obtained if the treatment with alkalis is effected with solutions of less than 1% concentration.

A. J. Hall.

Improving artificial fibrous materials. L. Lilienfeld (E.P. 253,854, 17.7.25. Conv., 17.6.25. Addn. to 231,806; B., 1925, 985).—Artificial silk (viscose, cuprammonium, Chardonnet, and cellulose acetate) yarn in skeins or fabric is impregnated with a solution of a cellulose thiourethane in which at least one hydrogen atom of the amino-group is replaced by an alkyl radical, the solvent being subsequently removed; the resulting silk has 40—100% increased tensile strength. Suitable solvents include aqueous solutions of alkalis such as caustic soda or ammonia, and volatile solvents such as pyridine. The silk yarn is preferably stretched during the treatment (cf. E.P. 253,853; preceding), and the impregnated silk may be rendered highly flexible by exposure to the vapours of a suitable organic solvent, *e.g.*, pyridine, as described in the chief patent and also in E.P. 248,994 (B., 1926, 532). For example, viscose yarn is drawn in a stretched condition through a solution containing 100 pts. of a cellulose-*N*-phenylthiourethane prepared as described in the chief patent or E.P. 248,994 or 248,246 (B., 1926, 400), 900 pts. of 2% caustic soda, and 2000—6000 pts. of water, then passed through a precipitating bath containing 10% of sulphuric acid, washed, and dried; after-steaming is optional.

A. J. Hall.

Manufacture of artificial silk. Courtaulds, Ltd., and C. M. Whittaker (E.P. 254,531, 27.7.25).—The difficulty of ensuring that the whole of the silk in any batch of viscose silk has a uniform affinity for dyes may be overcome by treating a number of skeins in the same dye-bath for the same time (using a dye that tends to produce uneven shades and is at the same time easily removable), sorting those skeins which show substantially the same shade into separate batches and finally removing the dye. The preliminary dyeing should be carried out at a comparatively low temperature to enhance any unevenness of shade. (Cf. Wilson and Imison, J.S.C.I., 1920, 323 T.)

D. J. Norman.

Artificial [silk] fibre and process of manufacture. R. Allwater and A. Heinemann (E.P. 255,623, 4.7.25).—Fibres are manufactured by the usual spinning process from a cellulose solution prepared by dissolving a protein such as keratin, fibrin, spongin, or konchoelin in a solution of a caustic alkali and adding successively cellulose and carbon disulphide and a further quantity of alkali if necessary to maintain the alkali in excess. The most suitable protein is keratin obtained from feathers, hair, horn, or hoof. A satisfactory spinning solution is prepared by dissolving 100 pts. of keratin in a 10% solution of caustic soda, adding 100 pts. of cellulose and 10—30 pts. of carbon disulphide, sufficient alkali being then added to maintain the product alkaline. The resulting fibres have greater resistance to water and absorb dyes more readily than viscose silk and to some extent have the properties of natural silk.

A. J. Hall.

Manufacture of artificial [coating, sizing, etc.] materials. L. Lilienfeld (E.P. 247,223, 8.2.26. Conv., 6.2.25. Addn. to 231,805; B., 1925, 985).—Derivatives of aniline are used instead of the bases proposed in the chief patent as solvents for cellulose-xantho-fatty acids in the presence of water since, with the exception of pyridine, the previously proposed bases yield solutions which become gelatinous on keeping. Suitable derivatives of aniline include the monoalkyl-derivatives or their homologues, and usually not more than 1 mol. of the base is sufficient to dissolve 1 mol. of cellulose-xantho-fatty acid, the resulting solutions being stable at room temperature for months. Although turbid solutions are obtained when an excess of a base is used, the stability of such solutions is good when derivatives of aniline are used as bases. The clear solutions dry to products which are insoluble in water and the resistance of which to water may be increased by heating or steaming. A solution suitable for conversion into artificial materials or for use as a coating material is obtained by kneading 100 pts. of air-dried cellulose-xanthoacetic acid (prepared as described in the chief patent) with 1,900 pts. of water containing 23 pts. of monomethylaniline. A. J. Hall.

Process of making cellulose acetate. W. R. Webb and C. J. Malm, Assrs. to Eastman Kodak Co. (U.S.P. 1,591,590, 6.7.26. Appl., 7.10.25).—Cellulosic material is treated in the presence of chlorine with an acetylating agent comprising a homogeneous mixture of acetic acid, acetic anhydride, and red phosphorus. D. J. Norman.

Cellulose acetate composition. B. K. Brown and C. Bogin, Assrs. to Commercial Solvents Corp. (U.S.P. 1,591,652, 6.7.26. Appl., 14.10.25).—The composition contains cellulose acetate and a multivalent metal salt of a monoalkyl ester of phthalic acid. D. J. Norman.

Chemical pulp-cooking process. G. Sivola (U.S.P. 1,590,987, 29.6.26. Appl., 4.12.25).—The progress of the digestion of sulphite-pulp is ascertained in the following manner:—A sample of the liquor is removed and its content of total sulphur dioxide determined. Simultaneously, a sample of pulp from the digester is washed, freed from water, and 2·5 g. (calculated on the dry product) are shaken with 200—300 c.c. of 0·01*N*-potassium permanganate at 25°, and the time taken for the solution to change from violet to yellow is noted. The speed of this reaction is a measure of the delignification of the pulp. These tests are repeated at half-hour intervals and the temperature is also taken. The results when tabulated enable the operator to decide on a temperature rise best suited to the economical production of a desired quality of pulp. R. B. Clarke.

Retting flax and other vegetable fibres. H. Thellier, Assr. to Soc. pour l'Appl. Ind. des Brevets Peufaillit (U.S.P. 1,594,389, 3.8.26. Appl., 26.12.24. Conv., 18.1.24).—See E.P. 227,836; B., 1925, 396.

Coating paper pulp vessels, plates, or the like for the protection thereof against the action of moisture, fat, soap, and the like. Baumgärtner, Katz & Co. G.m.b.H. (E.P. 241,876, 23.9.25. Conv., 27.10.24).

Electrodeposition of rubber on fabrics (U.S.P. 1,589,325).—See XI.

Electrodeposition of cellulose compounds (U.S.P. 1,589,326—8).—See XI.

Electrodeposition of mixtures of rubber and cellulose compounds (U.S.P. 1,589,320 and 1,589,322).—See XI.

Converting sulphite-cellulose waste liquors into tanning extracts (U.S.P. 1,592,062—3).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

New dyeing processes using Indigosol O. G. Friedländer (Textilber., 1925, 6, 916—917; cf. B., 1926, 705).—A full indigo shade four times faster to soap and also faster to rubbing than vat-dyed indigo is obtained directly by slop-padding fabric with Indigosol O and suitable assistants (cf. Indigosol DH, Vaucher and Bader, B., 1924, 864), particularly a large proportion of ammonium vanadate, and drying in a hot flue or even on drying cylinders. New effects are obtained by dyeing cotton with a mixture of Indigosol O and a suitable direct dye, *e.g.*, Diamine Pure Blue, and afterwards treating with iron or copper salts whereby the fastness to light of the resulting shade is improved; reserve effects are obtained with sodium thiosulphate, and both dyes may be discharged to white by means of a hyposulphite. A combination of Aniline Black and Indigosol O is being used in Japan. Brown grounds are produced with combinations of Indigosol O and Para Red or *m*-Nitraniline Orange. Fabric impregnated with a mixture containing 60 g. of Indigosol O, 5 g. of sodium thiosulphate, 223 g. of a solution of ammonium chlorate of 15° B. (*d* 1·115), and 712 g. of water, and dried at a low temperature, is sensitive to light and may be used for photographic reproduction. Indigosol O is more easily discharged than vat-dyed indigo by the usual chlorate discharge process. Fabrics having red and blue, or orange and blue, effects are produced by slop-padding the fabric with an Indigosol O preparation, drying, and then printing with a nitrosoamine red (*p*-nitroaniline) or orange (*m*-nitroaniline) reserve. Fabric printed with Indigosol O and aluminium, chromium, or iron mordants and afterwards steamed may then be dyed with alizarin or nitrosonaphthol. A. J. Hall.

[Prevention of] bronzy shades in dyeing with sulphur dyes. W. Kosche (Textilber., 1925, 6, 665).—The production of undesirable bronzy shades characteristic of the dyeing of cotton with black and blue sulphur dyes, particularly when a standing bath is used,

may be prevented by treating the dyed fabric with a 0·1% solution of sodium sulphide at 30°, or preferably with a solution containing 3% of acetic acid and 2% of sodium dichromate at 60°. Fabrics dyed with blue sulphur dyes and subjected to oxidation with a 1% solution of sodium perborate at 60° to develop the full shade are treated with sodium sulphide before the oxidation. Bronzy fabrics which have been dried are directly treated with a 0·2% solution of sodium sulphide, previous wetting-out being unnecessary. A. J. HALL.

[Prevention of] bronzy shades in dyeing cotton and artificial silks with sulphur, basic, and direct dyes. C. KOOX (Textilber., 1926, 7, 543—544; cf. Kosche, preceding abstract).—For removing the bronzy appearance of cotton and artificial silk fabrics dyed with sulphur dyes, the dyed fabric is treated with the usual hot "brightening" bath (containing a Monopole soap or oil and lactic, formic, or acetic acid) to which 1—2 kg. of gelatinised potato starch is added per 60—100 kg. of dyed fabric. The resulting non-bronzy fabric is somewhat harsh in handle, but this defect is removed by treating the material afterwards in a solution at 35—40° containing lactic, formic, or acetic acid and diastase (100 g. per 10 kg. of dyed material), and then washing it free from the starch hydrolysed by the diastase. Bronzy shades on artificial silk dyed with basic dyes are avoided by the addition of potato starch (50—100 g. per 10 kg. of dyed material) to the dye-bath and afterwards correcting the resulting harshness and loss of brilliancy of shade by treatment with a solution of diastase; the resulting shade has increased fastness to rubbing.
A. J. HALL.

Influence of after-treatments on the fastness to light of dyeings obtained by means of Naphthol AS compounds. E. KAYSER (Textilber., 1926, 7, 437—440).—Cotton dyed with Naphthol AS–SW and Fast Red KB base contains the resulting yellowish-red pigment uniformly distributed throughout the cell wall, the cuticle and lumen being free from pigment, but during subsequent soaping for ½ hr. at boiling point the pigment becomes crystalline and bluish-red, and the greater part migrates to within the lumen. This behaviour, which confirms the observations of Haller and Ruperti (B., 1926, 316), is more readily seen in fibres swollen by means of caustic alkalis or cuprammonium solution, so that the cell wall (cellulose) occupies about two-thirds and the lumen one-third of the fibre. Migration of pigment does not occur in boiling water or boiling (*ca.* 109°) solutions of sodium chloride, but does occur in boiling water containing an alkali and also to a limited extent in solutions of soap containing sodium chloride. Migration is dependent on swelling of the cotton cellulose and is accompanied by an increase of fastness of shade to light. For example, shades produced on cotton by means of Naphthol AS and Fast Red GL base, and Naphthol AS–SW and Fast Red KB base, afterwards washed in cold water and soaped at 50°, 75°, and boiling point showed increasing fastness to light. Photomicrographs of sections of dyed fibres before and after soaping are given. Changes of shade produced by after-treatment are due to optical effects and not to change of constitution of the dye. A. J. HALL.

Leather dyeing. III. H. SALT (J. Soc. Leather Trades Chem., 1926, 10, 168—171; cf. B., 1926, 153).—Basic dyes give full shades on chamois leather, but are not readily absorbed and retained if the leather is re-tanned with chromium salts. Unchromed chamois leather will retain 1% of basic dye in the surface layer. The basic dye diffuses evenly through chromed chamois. Direct dyes alone are of little use in dyeing chamois leather. Acid dyestuffs will dye chamois leather whether re-tanned with chromium salts or not. It is suggested that both the basic dyes and the chromium compounds combine with the acid groups in the hide substance molecule. Hence the chamoising must affect these groups very little, if at all. Chroming of chamois leather does not affect the dyeing with acid dyes, hence the chromium salts cannot combine with the free basic groups in chamois leather. D. WOODROFFE.

Leather dyeing. IV. H. SALT and A. ASTROM (J. Soc. Leather Trades Chem., 1926, 10, 197—199; cf. preceding abstract).—Chrome leather was dyed with 2% of Primuline extra, Chlorazol Brown G.M., or Chlorazol Brown M., without the application of tannin, then washed and drummed in 500% of water (on weight of stock) containing 4% of hydrochloric acid and 2% of sodium nitrite for 20 min. at the lowest possible temperature. After rinsing, the dyed leather was transferred to a solution of 0·3% of phenol, resorcinol, α- or β-naphthol, and 0·1% of caustic soda or a solution of 0·3% of *m*-phenylenediamine and 0·14% of soda ash. This coupling process changed the colour, but it was fast to water, hot soap solution, sun, air, and 1% sodium carbonate solution. The dyed leather should be treated with 0·5% borax solution to neutralise any acid left from the diazotising process. D. WOODROFFE.

Reserves obtained by means of algin. KUNIG (Textilber., 1926, 7, 538—539).—"Algin," or "norgin," a product (containing alginic acid) obtained from seaweed, forms soluble sodium and magnesium salts, but with salts of alkaline-earth and heavy metals yields insoluble horn-like products which resist penetration by dye liquors, and may therefore be used in reserve pastes for printing and batik dyeing. Veined batik effects obtained by means of fabric prepared by impregnation with an 8—10% solution of algin, drying, coagulating with a suitable metal salt, washing, and drying, are exceptionally clearly delineated; fabric thus prepared is superior to that prepared in the usual manner with wax, since it may be calendered, heated, or steamed without reducing its resist properties. A particularly suitable method for preparing fabric with algin consists of impregnating it with an ammoniacal solution of copper sulphate containing algin and subsequently fixing the copper alginate in an insoluble form by steaming. Algin resists on fabrics are removed by successive treatment with an acid and a weak alkali, *e.g.*, sodium carbonate,

so that only dyes fast to these treatments may be used. A. J. HALL.

PATENTS.

Dyeing cellulosic fibres. DURAND & HUGUENIN, S.A. (E.P. 231,189, 20.3.25. Conv., 20.3.24).—More intense dyeings may be obtained on cellulosic fibres, particularly cotton, mercerised cotton, viscose, and the like, when using the ester salts of vat dyestuffs described in E.P. 186,057 (cf. U.S.P. 1,448,251; B., 1923, 398 A) if there is added to the dye-bath a salt, such as common salt or Glauber's salt, which lowers the solubility of the ester salt and thereby increases the proportion taken up by the fibre. *Example*: Viscose is immersed for 15 min. at the ordinary temperature in a bath containing per litre 6·6 g. of the sodium salt of the leuco-indigo sulphuric acid ester previously described (*loc. cit.*), 6·6 g. of sodium nitrite, and 33 g. of anhydrous sodium sulplate, the total weight of the solution being about ten times that of the fibre under treatment. The fibre is then wrung out and developed in twenty times its weight of a solution containing 2% of sulphuric acid (96%) and preferably the same proportion of sodium sulphate as was present in the dye-bath. The viscose is finally washed and dried. D. J. NORMAN.

Dyeing fast shades on wool. BADISCHE ANILIN- & SODA-FABR. (F.P. 601,430, 31.7.25).—Dyes not sufficiently soluble in water to allow of their direct application to wool from aqueous solutions are formed within the fibre by impregnating wool or half-wool materials with monohydroxy-compounds, except α- or β-naphthol, polyhydroxy-, amino-, or aminohydroxy-compounds and developing subsequently with suitable diazo-compounds. For example, brown (fast to milling), red (fast to milling and potting), and bluish-red shades are obtained by means of the sodium salt of 1 : 5-dihydroxynaphthalene and diazotised *m*-xylidine (in feebly alkaline solution), 2 : 3-hydroxynaphthoyl-7-hydroxy-2-naphthylamide and diazotised *p*-nitroaniline, and di-(2 : 3-hydroxynaphthoyl)-1 : 5-naphthylenediaminedisulphonic acid and diazotised *m*-xylidine respectively. 2 : 3 : 6-Benzamidonaphtholsulphonic acid and diazotised *m*-xylidine, di-(acetoacetyl)-*o*-tolidide and diazotised aminoazobenzene or *o*-chloroaniline yield red, orange, and yellow shades respectively. Black and reddish-brown shades are obtained by dyeing wool with the monoazo-dyes prepared from α-naphthylamine and 1 : 8 : 5-aminonaphtholsulphonic acid, and 5-nitro-2-amino-*p*-xylene and 2 : 8 : 6-aminonaphtholsulphonic acid, and then developing with diazotised acetyl-*p*-phenylenediamine or diazotised *m*-xylidine respectively. Deep black shades are obtained by dyeing wool with the monoazo-dye obtained from *o*-aminophenol-*p*-sulphonic acid and 1 : 5-dihydroxynaphthalene, then chroming and developing with diazotised *m*-xylidine. A. J. HALL.

Treatment of animal fibres with acid, alkaline, oxidising, or reducing liquors. M. BERGMANN, E. IMMENDÖRFER, and H. LÖWE (G.P. 426,624, 1.3.23).—Not less than 0·1% of saponin or a product containing saponin is added (as a protective colloid) to liquids capable of deleteriously affecting animal fibres. This use of saponin is applicable in the dyeing of wool with vat dyes from an alkaline hyposulphite vat, and in the carbonisation and chroming of wool. A. J. HALL.

Production of colour-tone and coloured effects on vegetable fibres. I. G. FARBENIND. A.-G., Assees. of G. RUDOLPH (G.P. 428,039, 23.9.23; Addn. to 348,530; cf. E.P. 173,313, B., 1922, 139 A).—Fabric woven from untreated yarn and yarn which has been mordanted with sulphurised phenols or their homologues and substitution derivatives, or condensation products of such phenols and aldehydes, or products obtained by treating sulphurised phenols with sulphites, is piece dyed. Alternatively, fabric woven from untreated and mordanted (with sulphurised phenols) cotton yarns is again mordanted in the piece with sulphurised phenols and then dyed with basic dyes, the twice-mordanted yarn then appearing darker in shade. A. J. HALL.

Dyeing cellulose esters, particularly cellulose acetate silk. I. G. FARBENIND. A.-G., Assees. of K. H. MEYER and H. HOPFF (G.P. 428,176, 1.3.24).—Cellulose acetate silk is dyed in shades fast to washing by means of slightly water-soluble, feebly basic nitroarylamines or their derivatives. Suitable orange-yellow, greenish-yellow, reddish-yellow, and yellow dyes are 8-nitro-2-naphthylamine, 3-nitro-4-aminobenzophenone, 4-nitro-2-aminodiphenylamine, and the condensation product of 3-chloro-6-nitroaniline and formaldehyde. A. J. HALL.

Dyeing cellulose silks, *e.g.*, viscose silks. E. O. SANNER (G.P. 428,263, 27.4.23).—Fabric consisting wholly or party of viscose silk is boiled in a solution containing 6% of Marseilles soap and 5% of an alcoholic soap solution (prepared by saponification of a vegetable fat in alcohol containing sodium carbonate), then bleached, hydro-extracted, and dyed in a boiling dye-liquor containing 6% of the alcoholic soap solution described above, and 2—3% of 85% formic acid added to the cold dye bath. The dyed material is withdrawn, hydro-extracted, and dried. The resulting fabric has a handle and lustre similar to those of natural silk. A. J. HALL.

Dyeing fast shades on animal fibres. I. G. FARBENIND. A.-G., Assees. of H. KRZIKALLA (G.P. 428,238, 24.6.23).—Shades fast to light and washing are obtained by dyeing wool in the usual manner for acid dyes with dyes produced by coupling arylides of 2 : 3-hydroxynaphthoic acid with diazotised aromatic compounds containing a sulphonic acid group. For example, a fast red dye is obtained from diazotised 2-chloro-5-toluidine-4-sulphonic acid and 2 : 3-hydroxynaphthoic acid. The dyeings are after-chromed when the dyes used contain suitable groups, *e.g.* the dye obtained from 6-nitro-2-aminophenol-4-sulphonic acid and 2 : 3-hydroxynaphthoic acid β-naphthalide. A. J. HALL.

Manufacture of fabrics [with pattern effects]. C. DREYFUS (E.P. 254,354, 6.2.25).—The process described

in E.P. 249,946 (B., 1926, 532) is applied in the production of pattern effects on fabrics composed wholly or partly of ethers or organic acid esters of cellulose.

D. J. NORMAN.

Production of discharges by means of hyposulphites. O. KUNZE (G.P. 426,024, 21.7.23).—Leather, skins, horn, celluloid, paper, and other materials susceptible to the action of heat and hyposulphites are treated with a discharge paste containing a hyposulphite, to which a substance of good heat conductivity may be added, and subjected to the action of acids in the presence of steam or gases at a suitable temperature; the discharge effect is thus completed in a short time at a temperature below 100°, and the material suffers no damage.

A. J. HALL.

Process of decorating material. A. Y. PEARL (U.S.P. 1,590,850, 29.6.26. Appl., 1.8.22).—A mother-of-pearl finish is produced on paper, celluloid, etc. by treating the material with ammonium alum solution, and then, while still moist, with ammonia fumes. The aluminium hydroxide film thus formed when dried is transparent, rendering the material iridescent. A dye may be dissolved in the alum solution. R. B. CLARKE.

Weighting silks. A. PEPPER (E.P. 256,479, 25.1.26. Conv., 14.8.25).—See U.S.P. 1,565,390; B., 1926, 123.

Producing multicolour effects on vegetable fibres. I. G. FARBENIND. A.-G., Assees. of J. RATH and W. CHRIST (U.S.P. 1,594,853, 3.8.26. Appl., 15.1.25. Conv., 7.2.24).—See E.P. 228,878; B., 1925, 845.

Dyeing apparatus [for hat bodies]. B. BÖHM (E.P. 248,359, 16.2.26. Conv., 25.2.25).

Devices for dyeing or similarly treating materials with liquids. C. H. HARTIG (E.P. 254,254, 29.12.25. Conv., 27.6.25).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Technology of the manufacture of water-glass [sodium silicate]. R. DECKERT (Chem.-Ztg., 1926, **50**, 535—536).—Sodium silicate is now prepared exclusively in regenerative gas-fired kilns similar to those used in glass manufacture, the working temperature being 1300—1400°. The wear on the furnaces is so heavy that the number in reserve must at least equal that in work. The sand, sodium sulphate, and sodium carbonate used must be perfectly dry, finely divided, and well mixed. The use of a greater proportion of sulphate than of carbonate, while giving a less alkaline silicate, renders the subsequent filtration easier. Examples of proportions recommended are given. The solid silicate, which must be kept free from organic impurities, is dissolved without crushing in rotating drums while steam is injected at 5—6 atm. When the solution reaches *d* 1·19 (cold) it is filter-pressed, the clear solution being usually somewhat coloured. Coloration can be minimised by washing the solid silicate with cold water in the drum before dissolving, or removed by filtration through blood charcoal, of which 500 g. are sufficient for the treatment of 10 tons of solid silicate. Sodium silicate solution of *d* 1·7 is prepared by adding caustic soda to the more dilute solution and concentrating *in vacuo*. As the concentrated solution cannot be filtered, purity of the initial solutions is essential. The final density reached varies with the proportion of Na_2O to SiO_2 in the solution. No use has so far been discovered for the filter-press residues from this process. C. IRWIN.

Electrolytic preparation of perborate. D. V. STEPANOV (Trans. Karpov Inst. Chem. [Russia], 1925, [4], 98—106; Chem. Abstr., 1926, **20**, 2288).—The electrolyte contained 30 g. of borax, 120 g. of anhydrous sodium carbonate, and 1 drop of alizarin and potassium dichromate per litre. For operation in presence of fluoride, which increases the current efficiency at the beginning of electrolysis, but later attacks the platinum anode with consequent catalysis of the decomposition of perborates, alizarin was replaced by naphthenic acids and sulpho-acids. The addition of sodium silicate checks the spontaneous decomposition of the perborate. High current density favours the formation of ozone, which does not contribute to the formation of perborate, but accelerates its decomposition, as also does rise of temperature. A current efficiency of 70—80% was attained. Since perborate is removed by crystallisation during the electrolysis, the addition, from time to time, of 0·382 g. of borax, 0·111 g. of sodium carbonate, and 0·167 g. of sodium hydrogen carbonate per litre is necessary.

A. A. ELDRIDGE.

Stability of bleaching powder. D. V. STEPANOV (Trans. Karpov Inst. Chem. [Russia], 1925, [4], 107—116; Chem. Abstr., 1926, **20**, 2393).—Bleaching powder prepared from lime calcined at 600° is unstable on account of the presence of carbonate, and the product is insufficiently chlorinated; the carbonates decompose at 930°. Calcination above 950° leads to an incompletely chlorinated product on account of the combination with silica. The best results on chlorination are obtained with slaked lime containing 4% of uncombined water. Traces of iron, manganese, nickel, or cobalt catalyse the decomposition of the bleaching powder, but the effect of iron is minimised by calcination of the lime at 950°. The presence of much air in the chlorination chamber is undesirable; the activity and stability of the product are inexplicably increased if the chlorine is first heated in a porcelain tube and then cooled.

A. A. ELDRIDGE.

Testing for the presence of barium chloride in calcium chloride. F. RICHARD (J. Pharm. Chim., 1926, [viii], 4, 49—53).—Calcium chloride solution, slightly acidulated with hydrochloric acid, is treated with a saturated solution of calcium sulphate and heated. On keeping, any barium or strontium present appears as a turbidity or precipitate. They may be distinguished

by treating the calcium chloride solution with a saturated solution of strontium chromate, when barium is precipitated as chromate, and may be weighed. Calcium chloride may be purified from barium by treatment with sulphuric acid, but will then contain traces of sulphate.
B. Fullman.

Manufacture of manganese sulphate, sodium nitrite, lead dichromate, and antimony salts. J. Milbauer [with L. Pick, O. Vogel, A. Meisl, J. Chloupek, B. Šlemr, and V. Judenič] (Chem. Listy, 1926, **20**, 389—396).—Manganese sulphate may be prepared from pyrolusite and ferrous sulphate by heating a mixture of equal parts of the two substances in a crucible above a layer of anhydrous ferrous sulphate at 700°. If the operation is carried out in an open furnace, serious losses of sulphur trioxide occur. The reduction of sodium nitrate to nitrite is best effected by heating the salt with an excess of 15% of granulated lead. The reaction commences at the m.p. of the nitrate, and, after 2 hrs. at 420°, complete reduction is obtained, but less lead than the theoretical amount is oxidised owing to a certain proportion of the salt being decomposed thermally with the evolution of oxygen. Lead dichromate, prepared by boiling the normal salt with concentrated chromic acid (Preis and Layman, Ber., 1880, **13**, 343), may be obtained in a pure dry form by washing the crystals with a mixture of 90% of acetone and 10% of glacial acetic acid. Very finely ground antimony dissolves fairly rapidly in solutions of tartaric acid, alkali tartrates, citric acid, and alkali citrates if a rapid current of air is passed through the solution, and much more slowly in lactic, hydrofluosilicic, and oxalic acids under the same conditions. An analytical method for the determination of nitrites, carbonates, formates, and oxalates in the presence of nitrates is suggested based on the reduction of aqueous solutions of nitrates by water-gas.
A. R. Powell.

Rapid method for the analysis of sulphur chloride. E. Benesch (Chem.-Ztg., 1926, **50**, 565).—50 c.c. of distilled water and 10 c.c. of 10% sodium hydroxide are placed in a 500 c.c. conical flask and about 0·5 g. of sulphur chloride, accurately weighed, is added, and a few drops of methyl-orange. The flask is warmed gently for 2—3 min. until all the sulphur chloride is decomposed, *i.e.*, until no oil remains on the bottom of the flask. Slight excess of 10% sulphuric acid is added, the mixture is heated to boiling for 10 min. to agglomerate the precipitated sulphur, a few more drops of methyl-orange are added, and after cooling, the liquid is diluted to 200 c.c. and 100 c.c. are neutralised with 0·2*N*-sodium hydroxide, and the chlorine is titrated with 0·1*N*-silver nitrate solution, using potassium chromate as indicator.
W. G. Carey.

See also A., Sept., 907, **Hydrolysis of aqueous solutions of sodium silicates** (Harman). 909, **Reduction of chromium sesquioxide and uranium dioxide with carbon, and action of nitrogen on uranium carbide** (Heusler). 915, **Action of nitric acid on metals in presence of catalysts** (Palit and Dhar). 916, **Formation of nitric oxide at high temperatures** (Briner, Boner, and Rothen); **Catalytic decomposition of sodium hypochlorite solutions by finely-divided metal oxides** (Chirnoaga); **Decomposition of potassium chlorate** (Burrows and Brown; Roginski and Schulz; Belenki). 917, **Catalytic oxidation of carbon monoxide** (Bray and Doss). 918, **Catalytic preparation of sulphuric acid** (Poliakov). 922, **Basic copper sulphates** (Fowles). 923, **Decomposition of alkaline-earth sulphates** (Zawadzki and others). 924, **Hydrolysis of sodium silicate** (Hägg). 927, **Determination of chlorine in perchlorates** (Dobroserdov and Erdmann). 928, **Determination of perchlorates** (Dobroserdov); **Determination of sulphurous acid and sulphites** (Alsterberg); **Reactions for carbonates, hydrogen carbonates, sulphites, and hydrogen sulphites** (Gaspar y Arnal).

Determination of arsenic. Evers.—See XX.

Patents.

Manufacture of concentrated and fuming sulphuric acid. Kudoh (F.P. 604,636, 29.8.25. Conv., 13.1.25).—A mixture of sulphur dioxide and oxygen is catalysed, the sulphur trioxide is absorbed with sulphuric acid, and the remaining gas is mixed with fresh sulphur dioxide and oxygen and again passed through the contact chamber.
W. G. Carey.

Production of highly concentrated commercial sulphuric acid. Metallbank u. Metallurgische Ges. A.-G. (G.P. 429,835, 1.3.24).—Gases containing sulphur dioxide are brought, in the presence of the necessary moisture and oxygen, into contact with highly concentrated nitrous vitriol, whereby a solution of nitrosylsulphuric acid is formed; from this an amount is abstracted corresponding to the daily output of sulphuric acid and is denitrated by means of gases poor in oxygen and containing more or less sulphur dioxide. The denitration may be assisted by heat, or may be completed by means of a reducing agent, such as hydrogen sulphide.
W. G. Carey.

Manufacture of hydrogen bromide. J. D. Riedel A.-G., Assees. of R. Hueter (G.P. 428,225, 11.1.23).—Pressure is maintained above atmospheric in the reaction chamber in the production of hydrogen bromide from its elements by ignition.
W. G. Carey.

Transforming barium and strontium sulphates into other barium and strontium compounds. F. Rothe and H. Brenek (E.P. 242,996, 11.11.25. Conv., 12.11.24).—Barium or strontium sulphate is mixed with silicic acid and heated to about 1200° in a rotary furnace in the presence of steam, in an oxidising atmosphere. The barium or strontium silicate thus obtained may be decomposed by acids to produce barium or strontium salts, and silicic acid which is used again in the process. It is advantageous to use the silicic acid in such proportions that the silicate produced

is of a type between M_2SiO_4 and M_3SiO_5. Such silicates when decomposed with water yield the alkaline-earth hydroxide, and the residue after treatment with water consists chiefly of the alkaline-earth metasilicate which is used again in the process in place of silicic acid.
H. ROYAL-DAWSON.

Manufacture of barium sulphide [and lithopone]. G. RICHARDSON (E.P. 255,167, 20.4.25).—Barium sulphate ground to 100-mesh or finer, is mixed with powdered carbon free from nitrogen, and formed into cylindrical briquettes with hollow cores, which are heated to redness in closed retorts. The barium sulphide produced is dissolved in water, and the solution may be treated with a solution of zinc sulphate to precipitate a mixture of barium sulphate and zinc sulphide (lithopone).
H. ROYAL-DAWSON.

Purification of barium silicates. C. DEGUIDE (G.P. 429,169, 9.4.25).—Barium silicate, with excess of silica, or alumina, is comminuted under water and treated with carbon dioxide, whereby the silica or alumina combined with the barium is set free in a gelatinous form, and is then removed from the barium carbonate or carbonate–silicate mixture by known methods, *e.g.*, in the form of soluble akali silicate and aluminate.
A. COUSEN.

Separation of barium chloride from solutions. RHENANIA VEREIN CHEM. FABR. A.-G., Assees. of F. RÜSBERG (G.P. 429,716, 6.10.23).—Sodium chloride is added with stirring and heating, and in such amount that a nearly saturated solution of sodium chloride remains.
W. G. CAREY.

Catalytic processes and apparatus for reactions between gases [*e.g.*, synthesis of ammonia]. SYNTHETIC AMMONIA & NITRATES, LTD., and F. H. BRAMWELL (E.P. 255,232, 17.7.25).—The gases, *e.g.*, for synthesis of ammonia, are caused to flow first downwards through a mass of the catalyst and then upwards through one or more narrow tubes embedded in the mass. Removal and replacement of the catalyst are facilitated by supporting it on a perforated grid spaced from the closed lower end of the container. The open lower ends of the tubes extend through the grid and their upper ends form part of or are connected with the inner tubes of a heat-exchanger. The gases are supplied to the upper end of the container through the outer tubes of the heat-exchanger.
H. HOLMES.

Catalytic synthesis of ammonia. S. G. S. DECKER. From H. HARTER (E.P. 255,278, 7.10.25. Addn. to 241,771; B., 1926, 12).—The method of combining high- and low-pressure synthesis with transfer of heat from the high-pressure chamber to the other has proved difficult to operate owing to the action of hydrogen on the chamber walls. The method is therefore modified by insulating the catalyst chamber walls, the temperature of which does not exceed 300—400°. Heat transference is then performed in a separate exterior heat-exchanger or by radiation from the moderately heated high-pressure catalyst chamber wall, or by both methods.
C. IRWIN.

Basic chromic salts and process of making them. H. RÖSSNER and E. RUNNE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,592,961, 20.7.26. Appl., 16.12.25. Conv., 8.11.24).—Solutions of chromic salts, containing per 152 parts by weight of Cr_2O_3 not more acid than is equivalent to 140 parts of SO_3, are treated in a finely divided condition with a stream of hot gas to obtain readily soluble basic chromic salts.
H. MOORE.

Recovering potassium chloride from brine. J. L. SILSBEE (U.S.P. 1,593,038, 20.7.26. Appl., 21.1.21). —A solution nearly saturated with potassium chloride is mixed with sufficient magnesium chloride, in a concentrated form, to cause separation of practically the whole of the potassium chloride.
H. ROYAL-DAWSON.

Solid calcium nitrate. BADISCHE ANILIN- & SODA-FABR. (F.P. 604,116, 3.10.25. Conv., 4.12.24).—A concentrated calcium nitrate solution is applied to a drum heated to 200°.
A. COUSEN.

Decomposition of gypsum. RHENANIA VER. CHEM. FABR. (F.P. 604,699, 30.9.25. Conv., 2.12.24).—Gypsum is mixed with silica or silicious material and heated to 1100° in steam and an inert gas.
W. A. CASPARI.

Manufacture of potassium hydroxide from crude potassium salts. M. ALLINGER (F.P. 604,702, 1.10.25). —Kainite, sylvinite, or a mixture of alkali sulphates is treated in concentrated solution with oxalic acid, and the precipitated oxalate treated with calcium hydroxide. The oxalic acid is regenerated from the calcium oxalate by means of sulphuric acid and the calcium sulphate is used, with ammonia and carbon dioxide, to prepare ammonium sulphate.
A. COUSEN.

Manufacture of alkali sulphates and glaserite. F. STEIN (F.P. 604,891, 20.10.25. Conv., 20.10.24 and 2.2.25).—Sodium sulphate or glaserite, potassium chloride, and water are heated together in such proportion that glaserite is formed and simultaneously the solution becomes saturated with potassium chloride and sulphate, so that these salts are precipitated.
W. G. CAREY.

Manufacture of sodium sulphate. W. MECKLENBURG (G.P. 430,092, 15.2.25).—The double salt of sodium and ferrous sulphates, prepared in the usual way, is treated with a solution of sodium chloride of suitable concentration.
W. G. CAREY.

Recovery of iodides from iodine adsorbed by charcoal. BOUW-MAATSCHAPPIJ ARINA (F.P. 604,989, 23.10.25. Conv., 16.4.25).—The charcoal is treated with a solution of alkali sulphite and washed with water.
W. A. CASPARI.

Direct transformation of kieserite into solid Epsom salts without heating. KALI-IND. A.-G., and

Ratig (G.P. 429,543, 3.2.23).—Kieserite is stirred with magnesium sulphate solution and during the reaction water is added in such amount that the mixture remains capable of being stirred. W. G. Carey.

Decomposition of magnesium chloride by steam. J. Kersten (G.P. 429,654, 15.11.24).—Carbon is mixed with the magnesium oxychloride in order that the steam passed over the mixture may be decomposed with production of nascent hydrogen. W. G. Carey.

Production of chromate solutions practically free from iron. H. C. Starck, Komm.-Ges. auf Aktien, F. Klaus, and R. Basler (G.P. 429,655, 12.8.23. —A solution of ferrochrome in sulphuric acid is mixed with potassium dichromate and free sulphuric acid, and boiled under atmospheric or higher pressure; if chrome alum is required, alkali sulphates are then added. W. G. Carey.

Preparation of alumina and its salts. I. G. Farbenind. A.-G., Assees. of H. Specketer, G. Münch, E. Marburg, and W. Handrich (G.P. 429,717, 24.6.21).—Acid sulphate solutions resulting from the decomposition of clay are treated for the reduction of the ferric iron, and the alumina is precipitated by means of alkalis, or ammonia, ammonium carbonate, or ammonium sulphate with or without sodium bicarbonate, the total alkali being slightly less than the quantity equivalent to the acid present, so that the precipitate consists of a basic aluminium sulphate free from ammonia, approximately $2Al_2O_3,SO_3$, which is converted in the usual way into alumina or its salts. In the case of acid chloride solutions, after reduction of the ferric iron, the basic aluminium sulphate is precipitated by adding sulphates, the precipitation being effected at an elevated temperature and under pressure. W. G. Carey.

Production of phosphorus in the electric furnace. F. Tharaldsen (Nor.P. 40,306, 16.1.23).—Ferrosilicon or silicon carbide is employed as the reducing agent in the preparation of phosphorus from natural phosphates in the electric furnace. W. G. Carey.

Sulphur burners. E. C. R. Marks. From Grasselli Chemical Co. (E.P. 256,080, 29.9.25).—See U.S.P. 1,566,538; B., 1926, 155.

Producing hydrogen and phosphoric acid. F. G. Liljenroth, Assr. to Phosphorus-Hydrogen Co. (U.S.P. 1,594,372, 3.8.26. Appl., 2.3.23. Conv., 28.8.22).—See G.P. 406,411; B., 1925, 242.

Continuous separation of sulphur from sulphur solutions. E. Legeler (U.S.P. 1,594,783, 3.8.26. Appl., 1.12.25. Conv., 7.6.25).—See E.P. 249,044; B., 1926, 406.

Burning of gypsum (G.P. 428,576).—See IX.

Effluents from fuller's earth and similar factories (G.P. 428,486).—See XXIII.

VIII.—GLASS; CERAMICS.

Temperature-sensitiveness of refractory materials in the glass industry. K. Endell and W. Steger (Glastech. Ber., 1926, 4, 43—57; Chem. Zentr., 1926, II, 480).—The authors define temperature-sensitiveness as equal to the quotient of the coefficient of thermal expansion by the torsional power at 500—600°. The thermal expansions of silica bricks and tank blocks, and the heat conductivity of a highly silicious tank block were determined. The tendency to splitting was determined by heating three bricks in a gas furnace upon one side only to 850°, and then plunging into water, the process being repeated until the brick broke. Quartzose bricks split after 1 to 3 quenchings, but some samples withstood over 15 tests. Bricks were temperature-sensitive only below 600°. A. Cousen.

Thermal expansion of fused silica. W. Souder and P. Hidnert (Sci. Papers U.S. Bur. Standards, 1926, **21**, [524], 1—23).—The expansion of 17 samples was determined, measurements being made over the range — 125° to + 1000°. Pointed cylindrical rods, 300 mm. long, were heated in an air- or gas-tube furnace at the higher, and in a bath furnace at the lower, temperatures. In the former case fine weighted wires hung vertically from the ends of the rod through slits in the chamber and movement of the wires was measured; in the bath chamber the wires extended upwards from finger supports beneath the ends of the specimen. Over the range — 125° to + 20° a minimum value was obtained at temperatures varying from — 50° to — 97°, coefficients at temperatures below the minima ranging from — 0·03 to — 0·30 ($\times 10^{-6}$), and, above, from + 0·12 to + 0·29 ($\times 10^{-6}$). In the higher ranges the following average values were found: 20—60°, $0·40 \times 10^{-6}$; 20—100°, $0·45 \times 10^{-6}$; 20—300°, $0·53 \times 10^{-6}$; 20—400°, $0·55 \times 10^{-6}$; 20—500°, $0·52 \times 10^{-6}$; 20—750°, $0·50 \times 10^{-6}$; 20—1000°, $0·48 \times 10^{-6}$. The coefficient of transparent samples was slightly larger than that of opaque ones, and rods annealed at 1000° gave slightly smaller values than unannealed samples. A summary of results by other observers is given. A. Cousen.

Physical properties of glasses. Relationship to chemical composition and mode of preparation. W. E. S. Turner (J.C.S., 1926, 2091—2116).

***X*-Ray examination of aluminium silicates.** Rosbaud and Mark.—See A., Sept., 889.

Patents.

Protective lenses. E. Busch A.-G. Optische Ind. (G.P. 424,811, 10.1.25).—As a protection against dangerous visible or ultra-violet rays, lenses are coloured by a suitable glaze, whereby the tint can be regulated as required. If desired, only the upper edge, through which the dangerous rays usually enter the eye, may be so treated. A. Cousen.

Electrofining glass furnace. W. G. Clark (U.S.P. 1,594,496, 3.8.26. Appl., 6.4.20).—See E.P. 161,192; B., 1922, 711 A.

Annealing glass. A. E. WHITE. From MISSISSIPPI GLASS CO. (E.P. 256,040, 17.7.25).

IX.—BUILDING MATERIALS.

Effect of the process of manufacture on the properties of calcined gypsum. L. E. SMITH (Rock Products, 1926, **29**, 56—59; Chem. Abstr., 1926, **20**, 2401).—A study of the effect of the time and temperature of calcination of gypsum on the tensile strength of admixtures with sand (1 : 3); maximal values are 195 min. and 197°. The plasticity of the mixtures increases with the fineness of the gypsum and with the hemihydrate content; it is decreased by artificial anhydrite and by silica (5%). The properties of the gypsum are not seriously affected by the presence of calcium carbonate (5%), magnesium carbonate (5%), ferric oxide (2%), magnesium sulphate (3%), sodium chloride (if $< 0\cdot125\%$), or sodium sulphate (if $< 0\cdot5\%$).

A. A. ELDRIDGE.

See also A., Sept., 899, **Setting of plaster of Paris** (NEVILLE). 923, **Microchemical examination of gypsum** (BUDNIKOV).

PATENTS.

Binding material for bricks. A. F. FABRE (F.P. 603,549, 4.8.25).—A bond for all kinds of bricks consists of a mixture of powdered gypsum and natural or artificial puzzuolana, to which sand may also be added, treated with solutions of caustic soda, potash, or baryta. Double sulphates of calcium and sodium, potassium, or barium, together with calcium or barium silicate, are formed, and setting follows with great rapidity.

A. COUSEN.

Burning of gypsum. MASCHINENBAU-ANSTALT U. DAMPFKESSELFABR. A.-G. DARMSTADT, VORM. VENULETH & ELLENBERGER, GÖHRIG & LEUCHS, and A. STEINBRÜCKNER (G.P. 428,576, 21.6.24).—Gypsum and similar material is burned in a rotary kiln in which the heating gases, obtained from liquid fuel, are blown with a spiral motion opposed to that of the kiln. For this purpose the kiln is provided with a conical ignition and combustion chamber to the inner wall of which are attached screw-shaped ribs.

A. COUSEN.

Refractory heat insulating material. A. J. H. HADDON. From CELITE CO. (E.P. 256,021, 27.6.25).—See U.S.P. 1,544,433; B. 1925, 718.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Agglomeration and briquetting of finely divided ferriferous material. M. OTTOLENGHI (Annali Chim. Appl., 1926, **16**, 237—268).—Descriptions of the various methods used in the working up of finely divided iron ores and residues are given including briquetting with and without binders, and sintering. Agglomeration can be performed by fixed or rotating furnaces, with hot or compressed air, and by roasting. A new process devised by Piccinini is described, which is a modification of the Huntington–Heberlein method, and consists in heating the fine material admixed with coal to incandescence. Hot air is then blown through the mass from below through channels provided for the purpose. A hard solid porous block is obtained which is easily broken up and in very suitable condition for treatment in the blast furnace.

S. B. TALLANTYRE.

Effect of occluded hydrogen on the tensile strength of iron. L. B. PFEIL (Proc. Roy. Soc., 1926, **A**, **112**, 182—195).—Tests were made on iron in the normal finely crystalline condition, on single crystals, and on the boundary between large crystals. Electrolytic pickling was used and the tests were made with the specimen actually immersed in the acid. The variables studied were the strength of the acid electrolyte and the electrolysing current, the time the current passed before beginning the loading, the diameter of the test-pieces, the rate of loading, and the temperature at which the tests were made. The influence of temperature is considerable, the decrease of tensile strength being less marked at temperatures slightly above air temperature. Occluded hydrogen has a considerable weakening effect on the intercrystalline boundaries both of large and small crystals, and decreases the cohesion across the cubic cleavage planes. In a single crystal, the tensile strength is hardly affected, but the appearance of the fracture is entirely altered. Experiments with the finely crystalline aggregate showed that the effect of the hydrogen only persists for a very short time, and unless the pickling is continued up to the actual breaking of the specimens, the effect is scarcely apparent. This indicates how limited in value is the tensile test for the investigation of failures in iron and steel suspected to be due to occluded hydrogen.

L. L. BIRCUMSHAW.

Determination of oxygen in iron. P. OBERHOFFER, J. KEUTMANN, W. HESSENBRUCH, and E. AMMON (Stahl u. Eisen, 1926, **46**, 1045—1049; cf. B., 1925, 993).—A.—By the hydrogen process. Improvements in the apparatus (cf. *loc. cit.*) are described, including the substitution of a Silit for the chrome–nickel furnace. The average of a large number of experiments shows that iron in the form of turnings contains $0\cdot024\%$ more oxygen than when in small pieces. The method in its limited sphere of application (absence of carbon and silica) is considered trustworthy. Contrary to the statement of Chaudron and Blanc (B., 1923, 58 A), higher amounts of manganous oxide than $0\cdot2\%$ can be reduced if sufficient time is taken. B.—By the hot-extraction process. An arrangement has been devised by which the gases can be drawn off in stages and the reduction followed more closely. The manganese–carbon alloy containing $4\cdot2\%$ C and $0\cdot45\%$ Mn is now used exclusively in 20—30 g. amounts for each determination. Good results are always obtained if oxides of iron only are present, but with manganous oxide and silica the reduction is incomplete. This is the case with samples deoxidised with manganese and silicon. The method in its present form gives

results for the state of deoxidation which compare well with those obtained by the hydrogen process and are of the order expected when silica is present. The presence of oxygen in a steel is indicated by small areas of hardness, tendency to hard fissures, besides bad rolling and forging properties. C.—By the residue process. Except with synthetic iron-oxygen alloys, the bromine process (cf. *loc. cit.*) has not fulfilled expectations. If manganese is present the results do not show agreement. Morfitt's process (J. pr. Chem., 1854, **61**, 33) gives low results for ferrous iron. The modified iron chloride process of Troilus (Jernk. Ann., 1884, **39**, 432) is superior, but does not allow of the determination of manganese. The improved iodine process of Eggertz (Polytechn. J., 1868, **188**, 119) is more promising. The results of these processes for the determination of silica show satisfactory agreement with those of the chlorination process. It is impossible to determine iron at the same time as silica by the latter process, the values found decreasing with rising temperature. A series of analyses of poor and good tool steels by the recently described residue process, using bromine, is described. The results show good qualitative agreement. Curves are given showing the effects of lower temperatures and lower bromine concentration on the results. A. Coulthard.

Nature of the A1 transformation in carbon steels. K. Honda (Anniv. Vol. H. Nagaoka, 1925, 95—106; Chem. Abstr., 1926, **20**, 2136).—*X*-Ray analysis has established the author's theory that the A1 transformation in steel consists of the stepped change: austenite $\rightleftarrows$ martensite $\rightleftarrows$ pearlite or, more exactly, austenite $\rightleftarrows$ martensite $\beta \rightleftarrows$ martensite $\alpha \rightleftarrows$ pearlite. The step austenite $\rightleftarrows$ martensite is equivalent to the A3 transformation except for the presence of dissolved carbon. It is considered that in other alloys stepped transformations will take place for all eutectoid changes. The eutectoid transformation in aluminium bronze at 580° and the ageing of duralumin are specifically discussed from this point of view. A. A. Eldridge.

Rapid volumetric determination of large quantities of manganese in technical iron alloys. T. Heczko (Z. anal. Chem., 1926, **68**, 433—461).—The method depends on the oxidation of manganese to the tervalent form by heating the phosphoric acid solution of the alloy with permonophosphoric acid (cf. B., 1925, 430). The approximate manganese content is first determined by a rough test; 0·275 g. of alloy is dissolved in 7 c.c. of 83% phosphoric acid and the hot solution is treated with 2 g. of sodium pyrophosphate and 25 c.c. of permonophosphoric acid solution and, after boiling for 1—3 min., cooled, and titrated till colourless with 0·1*M*-thiosulphate solution. The true manganese figure is about 103% of the result obtained. For the final test, 0·55 g. is dissolved in 14 c.c. of 83% phosphoric acid; the hot solution is diluted with 25 c.c. of water or, if the alloy contains less than 10% Mn, with 15 c.c. of water and 10 c.c. of 0·*M*-manganese sulphate solution, and boiling continued with the addition of ammonium persulphate until decomposition is complete and the added water expelled. The solution is treated with hydrogen peroxide until it becomes colourless, then with a crystal of ferrous ammonium sulphate to remove excess of the peroxide, 4 g. of sodium pyrophosphate and 6 c.c. more than the theoretical quantity of permonophosphoric acid (calculated from the rough test) are added, and the solution is heated gently over a small flame for 3 min. to decompose excess of the oxidising agent. After cooling and diluting, potassium iodide is added and the liberated iodine titrated with thiosulphate. The permonophosphoric acid is made by mixing 55 g. of phosphorus pentoxide with 12 c.c. of perhydrol in a beaker immersed in ice and diluting to 1 litre; 1 c.c. of the solution oxidises 1% of manganese using 0·55 g. of alloy. Chromium, when less than about 1%, is determined simultaneously with the manganese; larger amounts give erratic results. Much cobalt interferes with the colour of the end-point, and vanadium leads to high results if more than a small proportion is present. For good results for manganese exact adherence to the above directions is essential. A. R. Powell.

Palladium alloys in jewellery and their detection. F. Durdik (Chem. Listy, 1926, **20**, 406—407).—Palladium is often one of the constituents of the white gold alloys used in jewellery. To detect the palladium in these alloys the metal is rubbed on the usual touchstone and the white streak moistened with a mixture of equal parts of nitric acid (*d* 1·3) and hydrochloric acid (*d* 1·12). The liquid is then absorbed by means of a paper that has been previously soaked in an alcoholic solution of benzoylmethylglyoxime and dried; a light yellow spot on the paper indicates the presence of palladium (cf. Hanus, Jilek, and Lukas, A., 1926, 141). A. R. Powell.

Constitution and age-hardening of some ternary and quaternary alloys of aluminium containing nickel. (Miss) K. E. Bingham (Inst. Metals, Sept., 1926. Advance copy, 17 pp.; cf. B., 1923, 358 A).—The alloys of aluminium with magnesium and silicon possess the property of age-hardening after quenching from a high temperature, as do those of copper with aluminium to a less degree. The ternary alloys of aluminium with copper and nickel do not show this property. This is explained by the constitution of these alloys. Age-hardening is caused by the precipitation of $CuAl_2$, the solubility of which, both at high and low temperatures, is increased by the addition of 2% of nickel. Age-hardening occurs in these alloys when 1% of magnesium is added, due to the precipitation of Mg_2Si, the silicon being present as an impurity in the aluminium. C. J. Smithells.

Influence of gases on copper at high temperatures. I. A. G. Lobley and D. Jepson (J. Inst. Metals, 1926, **35**, 213—219).—See B., 1926, 279.

Hardness of cold-rolled copper. S. L. Hoyt (J. Inst. Metals, 1926, **35**, 231—257).—See B., 1926, 325.

Soft soldering of copper. T. B. Crow (J. Inst. Metals, 1926, **35**, 55—70).—See B., 1926, 325.

Determination of zinc oxide in brass. B. S. EVANS and H. F. RICHARDS (J. Inst. Metals, 1926, **35**, 173—180).—See B., 1926, 279.

Brittle ranges of bronze. W. L. KENT (J. Inst. Metals, 1926, **35**, 45—53).—See B., 1926, 326.

Mechanical properties at high temperatures of an alloy of nickel and copper. H. J. TAPSELL and J. BRADLEY (J. Inst. Metals, 1926, **35**, 75—105).—See B., 1926, 280.

Copper-rich aluminium-copper-tin alloys. D. STOCKDALE (J. Inst. Metals, 1926, **35**, 181—212).—See B., 1926, 279.

Die-casting of aluminium alloys. G. MORTIMER (J. Inst. Metals, 1926, **35**, 371—405).—See B., 1926, 326.

Corrosion of an ancient tin specimen. C. O. BANNISTER (J. Inst. Metals, 1926, **35**, 71—74).—See B., 1926, 327.

Constitution of the alloys of silver and tin. A. J. MURPHY (J. Inst. Metals, 1926, **35**, 107—129).—See B., 1926, 278.

Interpretation of macro-structure of cast metals. R. GENDERS (J. Inst. Metals, 1926, **35**, 259—293).—See B., 1926, 327.

Crystal growth in recrystallised cold-worked metals. W. FEITKNECHT (J. Inst. Metals, 1926, **35**, 131—172).—See B., 1926, 366.

Softening of strain-hardened metals and its relation to creep. R. W. BAILEY (J. Inst. Metals, 1926, **35**, 27—43).—See B., 1926, 366.

Production of single crystals of metals and some of their properties. H. C. H. CARPENTER (J. Inst. Metals, 1926, **35**, 409—438).—A lecture delivered on May 19, 1926.

Striation due to working or to corrosion in microscopical metallography. Mode of action of etching reagents. A. M. PORTEVIN (J. Inst. Metals, 1926, **35**, 363—370).—See B., 1926, 327.

See also A., Sept., 893, **Thermal study of electrolytic lead** (TRAVERS and HOUOT). 895, **Diffusion of solids** (HENRY). 896, **Relation between colour and structure of alloys** (CHIKASHIGE and others); **Separation of supersaturated mixed crystals** (FRAENKEL, SCHALLER, and QUINCKE); **Formation of deformation twins in eutectics** (VOGEL); **Influence of small additions of lead to gold** (NOWACK); **Behaviour of platinum metals towards silver and gold during cupellation** (TRUTHE); **Limits of resistance of tungsten-molybdenum mixed crystals** (VAN LIEMPT). 897, **Change of volume of cast iron on solidification and diagram of system iron-carbon** (HONDA and ENDO). 908, **Recurrent transition curves in anisotropic binary systems** (WEVER, GIANI, and REINECKEN). 909, **System iron-silicon-chromium** (DENECKE). 926, **Action of solutions of salts on ferrous metals** (GIRARD). 931, **Device for arc-welding easily oxidisable wires** (COATS).

Corrosion of iron water mains. HICKETHIER.—See XXIII.

PATENTS.

Process of making alloy steel. A. KISSOCK (Re-issue 16,396, 27.7.26, of U.S.P. 1,300,279, 15.4.19. Appl., 4.6.26).—See B., 1919, 504 A. The process is applicable to the production of molybdenum steel, the molybdenum being introduced as calcium molybdate.

Heat-treating magnesium alloy. Z. JEFFRIES and R. S. ARCHER, Assrs. to AMER. MAGNESIUM CORP. (U.S.P. 1,592,302, 13.7.26. Appl., 18.7.22).—A supersaturated solid solution of magnesium and aluminium is hardened and otherwise improved by being heated at a temperature of 125—300° for a sufficient period of time to cause precipitation of a hard constituent in a finely-divided state. S. S. WOOLF.

Process of recovering nickel. W. S. LIENHARDT, Assr. to METAL & THERMIT CORP. (U.S.P. 1,592,306—7, 13.7.26. Appl., 16.7.25).—To a solution of nicke containing also sulphuric acid which is to be subjected to cementation an additional reagent is added, viz., (A) a chloride ion, or (B) a sulphate of a metal not precipitated by iron. B. M. VENABLES.

Production of pure alkali metals. SOC. D'ÉLECTRO-CHIMIE, D'ELECTRO-METALLURGIE, ET DES ACIÉRIES ÉLECT. D'UGINE (F.P. 603,825, 27.12.24).—Hydroxides, fluorides, cyanides, carbonates, nitrates, or suphates of the alkalis are mixed with iron (preferably reduced iron) and heated, in the highest possible vacuum, above the m.p. of the alkali compound, whereupon the pure alkali metal distils in a yield of 70—80%. Chlorides, bromides, and iodides, because of the volatility of the resulting iron compound, are unsuitable. A. COUSEN.

Manufacture of chromium. J. H. BEAUMONT. From METAL RESEARCH CORP. (E.P. 256,433 14.10.25).—See U.S.P. 1,581,698; B., 1926, 548.

Applying protective layers on metls or other electric conductors. J. H. MELLQUIST (U.S.P. 1,595,675, 10.8.26. Appl., 1.8.24. Con., 23.8.23).—See E.P. 220,944; B., 1925, 886.

Heat treatment of metal castings. H. BARON. From E. WILL (E.P. 239,218, 27.8.25).

Construction of open-hearth furnace walls. OPEN-HEARTH COMBUSTION CO., Assees. of F NAISMITH (E.P. 242,607, 13.10.25. Conv., 10.11.24).

Furnaces [for reheating etc.] A. SMALLWOOD and J. FALLON (E.P. 255,938, 29.4.25).

XI.—ELECTROTECHNICS.

Graphic representation of electrolytic data. R. NITZSCHMANN (Chem.-Ztg., 1926, 50, 525).—It is shown how charts can be drawn up from which the relations between voltage, electrochemical equivalent, current efficiency, and energy consumption can be read off. Examples are given for the principal processes of technical electrolysis. W. A. CASPARI.

Dielectric breaking stress of liquids [transformer oils]. P. BARY (Rev. gên. Colloid., 1926, 4, 166—173).—Microscopical examination of spark gaps at 1000—3000 volts in transformer oils indicates that a dark-coloured filament is formed, under the dielectric stress, between the points. The filament, which is considered to consist of globules of asphalt suspended in the oil and surrounded by envelopes of water, takes a certain time to form in the briskly agitated oil lying in the gap, and a spark passes only when the gap is completely bridged. The material of the filament is then re-dispersed, but forms into line again after a few seconds, allowing another spark to pass. Defective insulating properties are attributed to the presence of asphaltic suspensions having aqueous envelopes, and the superiority of centrifuging to heating as a means of purification is adduced in support. W. A. CASPARI.

See also A., Sept., 892, **Temperature scale for tungsten filaments** (JONES). 893, **Physical properties of tungsten at high temperatures** (ZWIKKER). 913, **Electrolysis of chromium trioxide solutions** (MÜLLER). 919, **Electrochemical preparation of lead arsenates and arsenites** (ORMONT).

Electrolytic preparation of perborate. STEPANOV.—See VII.

Determination of arsenic. EVERS.—See XX.

PATENTS.

Packing for electrolytic apparatus for decomposition of water. R. PECHKRANZ (E.P. 237,903, 23.7.25. Conv., 2.8.24).—A resistant insulating packing is made of asbestos or other fibre impregnated with a hydrocarbon pitch such as bitumen from petroleum. W. A. CASPARI.

Heating coils for electric furnaces. BRIT. THOMSON-HOUSTON CO., Assees. of W. KEENAN (E.P. 245,089, 8.12.25. Conv., 27.12.24).—From the roof of a brick furnace, resistor coils are suspended by pins or hangers mounted in recesses in such a way that expansion or contraction is provided for. W. A. CASPARI.

Electric resistance elements for incandescent lamps and thermionic devices. GEN. ELECTRIC CO., and C. J. SMITHELLS (E.P. 255,195, 26.5.25).—A support for tungsten wire resistances working up to 2200° is made of magnesia with special precautions. The magnesia is sintered at 1800°, finely ground, mixed with a small quantity of unsintered material, moulded, and heated to 3300° or above, so that the surface is fused. W. A. CASPARI.

Electrodeposition of organic materials [rubber, etc.] on anodes from aqueous emulsions. C. L. BEAL and L. W. EBERLIN, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,589,324, 15.6.26. Appl., 7.5.25).—In the application of the process of U.S.P. 1,476,374 (B., 1924, 104), the harmful effects of nascent oxygen liberated at the anode (cf. U.S.P. 1,580,795; B., 1926, 639) can be avoided by forming the anode of, or coating it with, a metal which has a greater affinity for oxygen than the material being deposited. Alternatively, the metal is finely divided and dispersed with a protective colloid such as glue in the emulsion forming the electrolyte. Zinc, cadmium, and magnesium are suitable metals. The metal is oxidised preferentially to the coating, and the oxide is deposited with the latter. T. S. WHEELER.

Electrodepositing organic material such as rubber upon porous objects of non-conducting material such as fabrics. S. E. SHEPPARD and C. L. BEAL, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,589,325, 15.6.26. Appl., 1.4.26).—The depth and character of the deposit of organic material (rubber) obtained by the process of U.S.P. 1,476,374 (B., 1924, 104) upon a porous non-conducting fabric (cotton) may be controlled by first treating the material with a size containing a coagulant. Suitable sizes are starch, glue, or dextrin or similar hydrophile colloids adjusted to p_H 3—7 by an organic acid which acts as a coagulant. To ensure an even deposit the fabric is passed first through an alkaline degreasing bath containing a 5% solution of sodium carbonate, phosphate, or the like and then, after washing, led directly into the size. The sized fabric is then led around an anode formed of a roller of conducting material immersed in the electrolytic emulsion. T. S. WHEELER.

Electrodeposition of organic materials such as rubber and cellulose compounds. S. E. SHEPPARD and C. L. BEAL, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,589,326, 15.6.26. Appl., 22.4.25).—The deposits of organic materials on conducting surfaces obtained by the method of U.S.P. 1,476,374 (B., 1924, 104) are rendered more uniform by coating the surface of the conductor with a 3—5% aqueous solution of gelatin or glue, or with a dilute rubber emulsion, with which is preferably incorporated a hygroscopic substance, such as glycerol or calcium chloride, to inhibit hardening of the coating on storage. The coating forms a diffusion path for the current at the surface. T. S. WHEELER.

Electrodeposition of coatings of cellulosic compounds. Aqueous emulsions of electrodepositable cellulosic compounds and coalescing agents therefor. L. W. EBERLIN and C. L. BEAL, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,589,327—8, 15.6.26. Appl., 7.4.25).—The process of U.S.P. 1,476,374 (B., 1924, 104) can be applied to the electrodeposition of a cellulose

compound by dissolving the latter in a liquid which also functions as a coalescing agent and emulsifying the mixture in an aqueous solution with an emulsifier and a protective colloid. On passing a current through the mixture the cellulose compound is deposited on the conducting anode, the coalescing agent binding the deposited particles together. For example, cellulose nitrate or acetate, or a mixture of these compounds (500 pts.) dissolved in amyl acetate (1500 pts.), is emulsified with water (10,000 pts.) in a colloid mill in presence of an emulsifier, such as Turkey-red oil (150 pts.), and a protective colloid, *e.g.*, gum arabic (10 pts.). In the deposition a voltage of 110, and a current density of $\frac{1}{3}$ amp. per sq. in. of anode surface, is used for 4 min. If the substance to be coated is non-conducting, it is coated with a conductor such as graphite, or if it is porous, *e.g.*, wood, it is saturated with an aqueous solution of an electrolyte. T. S. WHEELER.

Electrodepositing rubber upon a metal wire. S. E. SHEPPARD and L. W. EBERLIN, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,589,329, 15.6.26. Appl., 20.11.25). —A metal wire is passed through a bath of the type described in U.S.P. 1,476,374 (B., 1924, 104) containing an aqueous emulsion of rubber, soap, sulphonated castor oil, and sulphur and if desired, aniline sulphate, and is thus coated with a mixture of rubber and sulphur. It is then led through a heater in which the rubber coating is partially dried, and after being passed between rollers which exert a firm uniform pressure on it and remove all inequalities in the coating it is led through a second heater in which drying is completed and the coating is vulcanised. T. S. WHEELER.

Aqueous emulsion containing electrodepositable rubber and a cellulose compound. Electrodeposition of coatings comprising rubber and a cellulosic compound. S. E. SHEPPARD and L. W. EBERLIN, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,589,330 and 1,589,332 15.6.26. Appl., 7.4.25).—An aqueous rubber emulsion of the type described in U.S.P. 1,476,374 (B., 1924, 104) and 1,589,331 (following), mixed with an emulsion of a cellulose compound prepared as described in U.S.P. 1,589,327—8 (preceding), is applied to the deposition of a mixture of rubber and a cellulose compound. T. S. WHEELER.

Aqueous emulsion of unvulcanised rubber and sulphur. S. E. SHEPPARD and L. W. EBERLIN, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,589,331, 15.6.26. Appl., 7.4.25).—A solution for use in the electrodeposition of rubber is formed from alkaline rubber latex, and sulphur emulsified with a protective colloid, such as glue or gum arabic. Accelerators, pigments, and fillers may be dispersed in the emulsion. The coating obtained is vulcanised after deposition. A suitable emulsion is formed from 4000 c.c. of 30% rubber latex and 1000 c.c. of a 20% emulsion of sulphur in 0·5% glue solution. T. S. WHEELER.

Utilisation of exhausted primary batteries. CHEM. FABR. JOHANNISTHAL, and SCHELLER (G.P. 428,267, 11.5.22).—The manganese-containing material from exhausted Leclanché cells is ignited in absence of air and the reduced zinc is removed. By treatment with dilute acid the graphite is separated, and the manganous oxide is then re-oxidised. W. A. CASPARI.

Recovery of lead compounds from old accumulators. F. FESSIA (G.P. 428,365, 27.2.23. Conv., 27.2.22).—The gratings of positive electrodes are freed from filling and used over again. Old fillings from positive and negative electrodes are subjected to heat treatment so as to produce a mixture of lead oxide and sulphate, which is applied to the filling of fresh electrodes. W. A. CASPARI.

Temperature-controlling devices for electric furnaces. BRIT. THOMSON-HOUSTON Co., LTD., and S. PAYMAN (E.P. 255,977, 11.5.25).

Phosphorus (Nor. P. 40,306).—See VII.

Purification of fats (F.P. 606,079).—See XII.

XII.—FATS; OILS; WAXES.

Crystalline bromides from linseed and other drying oils. H. TOMS (Analyst, 1926, **51**, 387—391; cf. B., 1924, 264).—The bromination products of perilla, candlenut, and Para rubber seed oils and the hexabromostearic acids isolated therefrom are identical with those obtained from linseed oil. The crystalline bromide from linseed oil (m.p. 156°) is not decomposed by prolonged boiling with ethyl acetate. Tetralin and acetone form a better crystallisation medium than ethyl acetate. Hexabromostearic acid was found on hydrolysing the bromoglyceride with 48% hydrobromic acid. D. G. HEWER.

Fixed oil of the seeds of "Kurrajong" (*Brachychiton populneum*. B.Br; syn. *Sterculia diversifolia*, G. Don). F. R. MORRISON (J. Proc. Roy. Soc. N.S.W., 1926, **59**, 267—275).—Two samples of seeds collected in 1924 and 1925 yielded, by ether extraction in a Soxhlet apparatus, 23·4% and 23·9% of oil, respectively, having m.p. 30°, below 15°; d^{30}_{30} 0·9083, d^{15}_{15} 0·9203; n^{20} 1·4676, 1·4709; acid value 65·0, 42·7; saponif. value, 198·0, 192·8; iodine value (Wijs, 2 hrs.), 101·3, 107·6; unsaponifiable matter (sitosterol), 1·1%, 1·5%. The fatty acids from the 1924 sample had m.p. 33·5°, d^{15}_{15} 0·8908, n^{40} 1·4548, iodine value (Wijs, 2 hrs.) 100·9, neutralisation value 202·4, mean mol. wt. 277, and consisted of palmitic, oleic, and linoleic acids. Alkaloids were absent from the oil. E. H. SHARPLES.

Constants of flax wax. W. HONNEYMAN (Pharm. J., 1926, **117**, 157—159).—Flax wax occurs principally in the fibre and cortical tissues of the plant, the air-dried cortex containing as much as 10%. Samples of wax from flaxes grown under different climatic conditions are shown to have practically identical characters. The analytical constants of flax wax and hemp wax are, respectively: saponif. value, 73·4—83·7, 101; iodine

value (Hanus, 2 hrs.), 21·6—28·8, 22·5; acid value, 17·5—23·8, 13·6; *d* 0·963—0·985, 0·977; m.p. 67·3—69·5°, 69·1°. C. O. Harvey.

Critical points of emulsification in oil-soap emulsions. E. V. Kyser and F. C. Vilbrandt (J. Amer. Pharm. Assoc., 1925, **14**, 392—398; Chem. Abstr., 1926, **20**, 2391).—Cottonseed oil, oleic acid, and various alkalis were employed. Small quantities of sodium or potassium carbonate produce permanent emulsions, separation being less likely or slower than if sodium hydroxide or soluble silicate is used. Sodium and potassium hydroxides are undesirable emulsifying agents. The viscosity of all the emulsions lay between 1 and 51·5, that of the oil used. A. A. Eldridge.

Change of refractive index of linseed oil during drying. Laurie.—See XIII.

Chaulmoogra oil. Gelarie and Greenbaum.—See XX.

Patent.

Refining of olive and other edible oils. E. Foray (F.P. 605,389, 17.10.25).—Free fatty acids are neutralised at 50° by sodium or potassium carbonate, and the oil is then treated with 0·1% of manganese dioxide and heated and stirred. H. M. Langton.

Purification of fats. G. Ravinetti (F.P. 606,079, 9.11.25. Conv., 18.11.24).—Oxidising gases (oxides of nitrogen) produced by electrical discharges are used, in a finely dispersed condition, shortly after their formation, for the purification of fats. H. M. Langton.

Manufacture of soap. M. I. Aische (E.P. 255,508, 17.3.25).—Soaps are made by saponifying soap-yielding materials, in presence of a hydrogenating agent, *e.g.*, an alkali amalgam, or metallic hydrides or alloys—with or without a catalyst, *e.g.*, reduced metals—or metal oxides, salts, or compounds. For example, a mixture of menhaden oil (566 pts.) and whale oil (565 pts.) is heated to 60° and emulsified with water at this temperature, and sodium amalgam in lumps (1348 pts.) is added gradually to the mixture. A vigorous reaction occurs, and after addition of all the amalgam the temperature is raised to 145—160° for a time. When saponification is complete the soap is remelted with addition of water, and salted out as usual; mercury from the amalgam settles out at the bottom of the vessel and is drawn off separately from the saline water and glycerol. H. M. Langton.

Presses for expressing oils or other liquids from materials containing the same. E. C. R. Marks. From F. S. Carver (E.P. 256,344, 16.5.25).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Analysis of Prussian blue. F. G. A. Enna (J. Soc. Leather Trades Chem., 1926, **10**, 172—174).—*Moisture*—1 g. of the finely powdered substance is dried for 2 hours at 105—110°, cooled, re-weighed, and dried for 1 hour more or till the weight is constant. Prussian blue should not contain more than 2·5—3% of moisture. *Total iron*—0·25 g. is treated for 15 min. with cold strong sulphuric acid, then heated over a very small flame for ½ hour until the acid just fumes. When the mass is white—not grey—it is rinsed into a flask with cold distilled water and heated until a clear solution is obtained. After filtering through a tared filter dried at 110°, any insoluble matter is washed, dried, and weighed. The filtrate is acidified with strong sulphuric acid, treated with pure granulated zinc, heated till colourless and no coloration is given with ammonium thiocyanate, diluted to 250 c.c. with freshly boiled distilled water, cooled, and made up to 250 c.c. 100 c.c. are titrated with 0·1*N*-permanganate; 1 c.c. = 0·012128 g. $Fe_7(CN)_{18}$. *Actual Prussian blue*—1 g. of the sample is treated with a hot 5% solution of hydrochloric acid, filtered, and the insoluble matter washed with boiling water till the filtrate gives no coloration with ammonium thiocyanate. The solution is reduced with zinc and sulphuric acid, the iron determined as before, and the result subtracted from the total iron found. The difference is actual Prussian blue. *Insoluble impurities*—The residue from the total iron determination is weighed and calculated as per cent. of insoluble matter. *Soluble impurities* is the difference of the sum of the above determinations from 100. D. Woodroffe.

Change of refractive index of linseed oil in the process of drying and its effect on the deterioration of oil paintings. A. P. Laurie (Proc. Roy. Soc., 1926, A, **112**, 176—181).—Possible causes of the lowering of tone of oil paintings with age are discussed, and this is shown to be due not only to the yellowing of the oil film, but also to a change in the refractive index. Considering pigments as consisting of transparent particles with the property of absorbing certain parts of the spectrum, it is clear that their apparent opacity and brilliancy will depend upon their refractive index and that of the medium with which they are mixed. By microscopical examination of a number of pigments in the three media, linseed oil, bromonaphthalene, and methylene iodide saturated with sulphur, it has been found possible to arrange the more important bright pigments in order of opacity. It has also been shown, by painting a film of linseed oil on the glass surface of a refractometer, that a considerable rise in the refractive index takes place during the drying of the oil, followed by a slow, continuous rise, which was followed for some months. As it is known that slow chemical changes, accompanied by an increase in density, continue in linseed oil for years, it is probable that the increase in refractive index will also continue. L. L. Bircumshaw.

Yellowing of oil films and change of refractive index of linseed oil on drying. A. P. Laurie (J. Oil and Colour Chem. Assoc., 1926, **9**, 163—173; cf. preceding abstract).—White pigments ground in various oil media were allowed to dry on glass and then kept in the dark under a bell jar containing a beaker of water for a period of 8 months, when their colour was compared with freshly prepared duplicates; fairly good matches were obtained with the yellow glasses alone of the Lovibond tintometer. The suggestion that unbleached

oils do not darken, and are therefore suitable for artists' colours, is not justified, as practically the same change of tint was observed in white pigments ground in dark yellow boiled oil and in refined linseed oil. Stand oil yellows appreciably less and walnut oil slightly less than linseed oil. Addition of resin which is not decomposed by heat during the process of dissolving, diminishes the yellowing of linseed oil, *e.g.*, the addition of mastic or dammar, but the reverse is the case for addition of copal varnish where the resin is decomposed.

S. S. Woolf.

Micrography of oil and varnish films. E. Stern (Kolloid-Z., 1926, **39**, 330—334).—An account of the microscopical examination of films of the binding media of paints and varnishes. The action of water on the films is studied. The results are to a considerable extent in agreement with the behaviour of the materials in practice, and it appears possible to develop the microscopical method to render the same service to the study of paints and varnishes that metallography renders to the study of alloys. A number of photomicrographs are given. N. H. Hartshorne.

Examination of resins. H. Wolff and W. Toeldte (Farben-Ztg., 1926, **31**, 2503—2505; cf. Eibner and others, and Schmidinger, B., 1926, 759, 760).—Tables are given indicating the nature and intensity of the characteristic fluorescences exhibited by various varnish resins in the form of lumps, powder, solutions in butyl acetate, and residue from evaporation of these solutions. In lump form the resins give irregular results, but inferences may be drawn from the fluorescence of powdered resins and from the change in appearance under the ultra-violet lamp on dissolving the resin and on evaporating the solvent. The Albertol resins give the most intense fluorescence (blue), whilst ester gum, lime-hardened resin, and Congo copal, in decreasing intensity, show light blue fluorescence. A marked increase in intensity is noticed when the acidity of resin is neutralised either by glycerol (ester gum) or by lime (lime-hardened resin). Combination of microscopical examination (Stock, B., 1925, 770), capillary analysis (Stock, B., 1926, 679), and fluorescence analysis will distinguish resins hitherto unclassifiable.

S. S. Woolf.

Storch–Morawski (Liebermann) reaction for detection of resin (abietic acid) or resin compounds in paints, etc. M. Schulz and F. Krämer (Farben-Ztg., 1926, **31**, 2556—2558).—Earlier statements to the effect that in the Storch-Morawski test for resin, a negative reaction alone is conclusive, while a positive reaction may possibly be due to constituents other than resin, are contradicted. Specifying a positive reaction to be the immediate formation of a violet coloration at least as deep as the tint of a 0·001*N*-solution of potassium permanganate, with rapid change to dirty brown or green colours, the authors declare that only in the presence of resin are these conditions fulfilled on applying the test. Erroneous deductions owing to the presence of phytosterol or of hydroxy-fatty acids, which give rise to colorations similar to those due to resin, are excluded by the above definition since the colour produced by the former is not the distinct violet required, while the latter retains a blue-violet colour for a too long period to be confused with the swiftly disappearing resin effect. As, however, it was found that oxidised resin does not give the reaction, a negative reaction does not necessarily prove the absence of resin initially. In using the gelation of abietic acid in benzene solution on addition of ammonia as a confirmatory test for resin, it must be borne in mind that fatty acids under this test give a similar although slightly less firm gel. S. S. Woolf.

Computation of colorimetric purity. I. G. Priest (J. Opt. Soc. Amer., 1926, **13**, 123—132). D. B. Judd (*Ibid.*, 133—154).

Reaction of "aluminon" with hydroxides of beryllium, rare earths, zirconium, and thorium. Middleton.—See A., Sept., 930.

Patents.

Stable pigment colours. W. Eberlein, and Colloisil Colour Co. (E.P. 254,887, 16.1 and 30.4.25).—Pigments, *e.g.*, primrose lead chromate, Prussian blue, that are of unstable nature when precipitated alone, are stabilised by mixing one or both of the components before precipitation with an organic or inorganic emulsion, colloidal solution or suspension, etc., or by precipitating or flocculating one of them with a fixing earth or clay. Typical stabilisers mentioned are bentonite, water-glass, and sodium–resin soap. S. S. Woolf.

Producing carbon black for use in the manufacture of rubber and rubber goods. H. H. Ward (E.P. 255,164, 20.4.25).—A mixture of finely-divided peat, cork, coal tar pitch, coal tar, and paraffin oil is formed into blocks and subjected to a regulated current of air in an externally heated furnace, *e.g.*, at 315°; the carbon black is collected by passing the resulting fumes through a series of chambers into which steam is injected. If no paraffin oil is used, the mixture is carbonised in the absence of air, the resulting charcoal or carbon being subsequently removed from the furnace and cooled in air-tight vessels. D. F. Twiss.

Vehicle for colouring matters and graphite. V. Lange, Assee. of H. Lange (E.P. 230,858, 16.3.25. Conv., 14.3.24).—A substitute for glue-media is obtained by boiling an aqueous solution of sugar, molasses, or syrup with a solution of asphaltum, gutta-percha, or similar product in benzine, benzol, etc., and removing the scum. S. S. Woolf.

Lacquers, impregnating materials, and the like. Farbenfabr. vorm. F. Bayer & Co. (E.P. 243,722, 23.11.25. Conv., 26.11.24).—The stability of solutions of cellulose acetate in glycerol- and other monochlorohydrins is improved by the addition of small quantities of organic bases, *e.g.*, carbamide or its derivatives, aliphatic amino-acids, aniline, pyridine, etc. A typical mixture is 10 pts. of cellulose acetate, 65 pts. of acetone, 20 pts. of ethylene chlorohydrin, 5 pts. of monchlorohydrin, and 0·5 pt. of glycine.

S. S. Woolf.

Resinous bodies from a phenol, furfural, and other aldehyde. C. ELLIS (U.S.P. 1,592,296, 13.7.26. Appl., 11.9.22. Renewed, 30.1.24).—Acetaldehyde is condensed with excess of a phenol in the presence of an acid catalyst. The readily fusible resin so formed is treated with sufficient furfuraldehyde to react with the excess of phenol, in the presence of a mild fixed alkali catalyst. S. S. WOOLF.

Dehydrating pine oil. R. C. PALMER, Assr. to NEWPORT Co. (U.S.P. 1,593,030, 20.7.26. Appl., 24.8.22). —Treatment of pine oil with a dehydrating agent yields a product more closely resembling turpentine than the original oil. S. S. WOOLF.

Manufacture of artificial resins. A. REGAL (E.P. 256,394, 29.7.25).—See U.S.P. 1,584,473; B., 1926, 596.

Lithopone (E.P. 255,167).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber—a fibrous material. F. KIRCHHOF (Kautschuk, 1926, 151—156).—Unvulcanised rubber which has been strongly stretched and then frozen in ice water resembles wool or "viscose" in its behaviour when further stretched; the stress–strain curve approaches the stretch-axis with increase in the degree of extension previous to freezing, or with more intense freezing. Relative to wool, the ultimate strength of the frozen stretched rubber is about one-tenth and its extension at break about three times as great. More intense freezing, *e.g.*, in liquid air, gives a product of greater strength. Both the crystalline portion of stretched rubber and the amorphous constituent are very sensitive to change of temperature. D. F. TWISS.

Aggregation and re-aggregation of raw rubber in the presence of other substances. M. KRÖGER (Gummi-Ztg., 1926, **40**, 2429—2430; cf. B., 1926, 137).—The re-aggregation of raw rubber in storage is less marked in samples with a higher content of natural "resin." The presence of a small proportion of a good carbon black reduces the rate of re-aggregation but a larger proportion accelerates it. Piperidine has a marked accelerating effect on the re-aggregation, whereas magnesium oxide retards it. D. F. TWISS.

Super-accelerators [for vulcanisation of rubber]. W. J. S. NAUNTON (Trans. Inst. Rubber Ind., 1926, **2**, 18—41).—A review of the present state of knowledge with respect to super-accelerators. These can be regarded as including salts (metal or organic) and disulphides of various dithio-acids. The metal salts are those of zinc or lead. Of the organic salts at least two find commercial use, viz., diethylammonium diethyldithiocarbonate, and piperidine piperidinecarbothionolate; the former of these is more active in the production of soft rubbers but less active for vulcanite. The diphenylguanidine salts of diethyldithiocarbonic acid, α-dithionaphthoic acid (salt, m.p. 154°), *iso*propylxanthic acid (salt, m.p. 105°), and mercaptobenzothiazole (salt, m.p. 172°), during vulcanisation in the presence of zinc oxide give the same results as a mixture of the free base with the zinc salt of the acid; with the third compound and corresponding mixture the effect is surprisingly less than that of an equivalent amount of zinc *iso*propylxanthate. Super-accelerators are not affected so adversely as less active substances, *e.g.*, diphenylguanidine, by the presence of certain other ingredients, *e.g.*, "substitute" or antimony sulphide, in the rubber mixture under vulcanisation. D. F. TWISS.

Production of coloured latex resistant to vulcanisation. R. DITMAR (Chem.-Ztg., 1926, **50**, 528—529).—A number of dyes have been examined as to their suitability for colouring latex which is subsequently to be dried and cold-cured. The following dyes pass a stringent test: yellow, pyramine yellow G extra, stilbene yellow 3 GX; red, sorbine red X, rhodamine B extra; pink, anthosine 5B; orange, cotton orange R; violet, vulcan red BN; blue, oxamine pure blue 6B. From 0·03% to 0·06% upon the latex is required to colour the goods. W. A. CASPARI.

Organic dyes in vulcanised rubber. T. J. DRAKELEY (Trans. Inst. Rubber Ind., 1926, **2**, 42—54).—In the presence of diphenylguanidine the accelerating influence of a neutral azo-dye is masked; Bismarck Brown has a more marked effect which, however, is not enhanced by zinc oxide. Organic dyes for introduction into rubber are most conveniently used after deposition on a filler such as clay. The dye should be sufficiently soluble in rubber to ensure even distribution but not so soluble as to cause transfer of the colour to adjacent rubber. The effect of vulcanisation, in moulds and in open steam, on the colour of rubber containing various dyes, is recorded. D. F. TWISS.

PATENTS.

Direct production of rubber goods from rubber emulsions. ANODE RUBBER Co. From P. KLEIN and A. SZEGVÁRI (E.P. 254,765, 13.2.25).—In the production of rubber goods from suspensions such as latex, difficulties may arise from certain features of the additional substances which it is desired to incorporate by previous dispersion in the latex. Such difficulties may be obviated by first forming an intimate mixture or compound of the desired ingredient with a second substance which is capable of masking the undesirable features of the primary substance. Thus liquid hydrocarbons may be satisfactorily dispersed in rubber latex if first impregnated in kieselguhr; lead oxide intimately mixed with kieselguhr gives a more permanent dispersion than lead oxide alone; sulphur may advantageously be introduced as zinc polysulphide or by first heating with kieselguhr, clay, lampblack, or unsaturated oils, and dispersing the powdered product in the latex. D. F. TWISS.

Accelerator for rubber vulcanisation. A. F. HARDMAN, Assr. to KELLY SPRINGFIELD TIRE Co. (U.S.P. 1,589,757, 22.6.26. Appl., 24.6.25).—A "master batch" of diphenylguanidine for vulcanisation purposes is produced by submitting a mixture of thiocarbanilide with an excess of metal oxide and a carrier to the action of ammonia under pressure. D. F. TWISS.

Treatment of rubber with aliphatic diamines and their derivatives. S. M. CADWELL, Assr. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,592,820, 20.7.26. Appl., 10.7.23).—Rubber is subjected to the reaction product of ethylenediamine and an open-chain aldehyde and is then vulcanised. D. F. TWISS.

Vulcanisation of rubber. H. O. CHUTE (U.S.P. 1,593,017, 20.7.26. Appl., 21.12.22).—Rubber is vulcanised with sulphur in the presence of a substance containing the characteristic carbon–nitrogen group present in calcium cyanamide. D. F. TWISS.

Rubber [vulcanisation] accelerator. G. H. BUCHANAN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,593,385, 20.7.26. Appl., 1.3.26).—A mixture of diphenylguanidine and ditolylguanidine is used for accelerating vulcanisation. D. F. TWISS.

[Rubber] adhesives. B. F. GOODRICH Co., Assees. of W. C. GEER (E.P. 247,136, 9.4.25. Conv., 9.2.25).—Rubber is converted into less chemically unsaturated derivatives of a tough balata-like nature. 7½ pts. of *p*-phenolsulphonic acid or similar product are mixed into 100 pts. of undissolved rubber on a mill, heated in an oven for 4—10 hrs. at 120—145°, the product worked on a warm mill to homogenise it and washed free of remaining reagent and other impurities. The balata-like product may be made by forming the sulphonic compounds in the rubber mass by adding to the raw rubber suitable organic compounds and sulphuric acid and heating the mixture. The balata-like product can be dissolved in benzene or other solvent to form a cement, or mixed with about 10% of an organic flux or softener, *e.g.*, tung oil, castor oil, hard mineral rubber, naphthalene, or Canada balsam, or with a relatively small proportion of a nitrogenous organic age-resister such as the thiocarbamide of dimethyl-*p*-phenylenediamine, 1 : 8-naphthylenediamine, aldol-α-naphthylamine, or benzidine. A suitable adhesive is prepared by mixing 86 pts. of the balata-like rubber product, 3 pts. of aldol-α-naphthylamine, 1 pt. of benzidine, and 10 pts. of tung oil. D. WOODROFFE.

Electrodeposition of rubber (U.S.P. 1,589,324—6, 1,589,329, and 1,589,331).—See XI.

Electrodeposition of mixtures of rubber and cellulose compounds (U.S.P. 1,589,320 and 1,589,322).—See XI.

Carbon black (E.P. 255,164).—See XIII.

XV.—LEATHER; GLUE.

Preparation of isoelectric collagen for tannin assay. L. MEUNIER and P. CHAMBARD (Rev. gén. Colloid., 1926, 4, 161—165).—Calf skin (250 g.) after treatment with lime to remove keratin, is well washed and agitated for several hours with 2½ litres of water containing 1·5 g. of acetic acid. After three washings with tap-water and a final one with distilled water, the p_H of the hide is about 7, but there is still some lime present. The hide is then soaked for six hours in 2½ litres of distilled water saturated with carbon dioxide, and the operation is repeated (about six times) until the extract no longer gives a reaction with ammonium oxalate. The hide should be preserved in water, which is saturated once a week with carbon dioxide; it contains about 0·1% (dry) of non-alkaline ash. When freed from dissolved carbon dioxide, the hide shows a point of minimum swelling, or isoelectric point, at p_H 5·4—5·6. W. A. CASPARI.

Determination of the isoelectric point of hide powder by means of complex chromium salts. K. H. GUSTAVSON (J. Soc. Leather Trades Chem., 1926, **10**, 203—211).—Portions of hide powder were respectively treated with solutions of acetato-complex chromium salts and formato-chromium complex salts of different p_H values. The p_H value of the exhaust liquor was determined and found to be slightly greater with solutions of $p_H < 5·04$, and slightly less with solutions of $p_H > 5·04$. It is inferred that the isoelectric point of hide powder is 5·0, though there is evidence to indicate that it may be a function of pre-treatment with acids and alkalis. D. WOODROFFE.

Fluorescence of the acetone extract of tanning materials. L. MEUNIER AND A. JAMET (J. Soc. Leather Trades Chem., 1926, **10**, 166—168).—Samples of coarsely ground tanning materials or the finely-powdered dry extract obtained by evaporating the aqueous infusion were shaken with 5 c.c. of acetone in a test-tube. Quebracho wood and Tizerah wood, as well as their dried extracts whether sulphited or non-sulphited, gave a characteristic yellow fluorescence, whilst chestnut wood, oakwood, myrobalans, valonia, divi-divi, and different kinds of galls showed a characteristic violet fluorescence when the acetone solution was examined in light from a Wood lamp. Mixed pyrogallol and pyrocatechol tans, *e.g.*, oak bark and gonakie, gave a bluish-white fluorescence. D. WOODROFFE.

Fluorescence of sulphite-cellulose extracts and its applications. L. MEUNIER AND A. JAMET (J. Soc. Leather Trades Chem., 1926, **10**, 212—213).—The pale violet fluorescence shown by solutions of sulphite-cellulose extracts can be used to detect adulteration in tannin extracts. 25 g. of sodium potassium tartrate are dissolved in 100 c.c. of water, and a 10% solution of normal lead acetate is added, drop by drop, with shaking until the precipitate at first formed re-dissolves. 15 g. of the tannin extract (*d* 1·190) are dissolved in 1 litre of water, 10 c.c. of the solution are mixed with 10 c.c. of the lead solution, a pinch of kaolin is added, and after filtering the clear filtrate is examined with a Wood lamp. A pale violet fluorescence will show with > 5% of sulphite-cellulose extract. Synthetic tannins cannot be distinguished from sulphite-cellulose extract by this means, but synthetic tannins are too expensive to be used as adulterants. D. WOODROFFE.

Properties of shoe leather. V. Area change with relative humidity. J. A. WILSON AND E. J. KERN

(J. Amer. Leather Chem. Assoc., 1926, **21**, 351—357; cf. B., 1926, 504, 556, 600).—Measurements have been made of the increase in area and water-content with increasing relative humidity of the atmosphere of the 18 leathers described previously. Vegetable-tanned leathers showed an average increase in area of 7% and chrome leathers an increase of 14·4% at 100% relative humidity as compared with zero relative humidity.

D. WOODROFFE.

See also A., Sept., 900, **Adsorption and swelling of hide powder** (KUBELKA and TAUSSIG). 936, **Organic chromium salts** (AGENO-VALLA and RAPOSIO).

Leather dyeing. SALT; SALT and ASTROM. See VI.

PATENTS.

Production of artificial inorganic-organic tanning materials, and tanning process. F. B. DEHN. From O. RÖHM (E.P. 255,313, 19.12.25; cf. U.S.P. 1,569,578, B., 1926, 206).—A tanning solution is made by adding salts of heavy metals, *e.g.*, ferric chloride or chrome alum, and alkalis or alkaline salts, *e.g.*, water-glass, to soaps and one or more suitable colloids, *e.g.*, sulphite-cellulose extract, in proportions to form in an acid solvent clear or colloidal solutions. H. HOLMES.

Conversion of sulphite waste liquors into tanning extracts. Preparing concentrated sulphite-cellulose extract containing magnesium compounds. W. E. B. BAKER (U.S.P. 1,592,062—3, 13.7.26. Appl., [A] 25.2.22, [B] 16.3.23.—(A) Sulphite-cellulose waste liquor is sprayed into the air to remove gaseous components and to oxidise other components. The product is then treated with an alkaline-earth oxide compound sufficient to decompose the reversible sulphurous acid present, filtered, and the concentrated filtrate treated with sulphuric acid to liberate the organic acids, which are tanning agents, and precipitate the alkaline-earth metal, which is then removed by filtration. (B) A concentrated solution of magnesium sulphate is used instead of sulphuric acid to precipitate the alkaline-earth metal.

D. WOODROFFE.

Manufacture of artificial horn from proteins or albuminous substances. F. SCHMIDT (E.P. 240,174, 18.9.25. Conv., 19.9.24).—To facilitate the kneading and pressing of the material, a neutral, readily volatile substance, such as ethylene chlorohydrin, ethyl lactate, glycol diacetate, alcohol, or acetone is added to the water used for the preliminary steeping. H. HOLMES.

Extraction process (G.P. 430,087).—See XX.

XVI.—AGRICULTURE.

Concentration of carbonates in two Minnesota soil types. P. R. MCMILLER (Soil Sci., 1926, **22**, 75—82).—Examination of the carbonate content of soils at varying depths indicated a definite zone of high carbonate concentration varying from about 18 to 30 in. in depth. The increase in carbonate from the surface downward is abrupt until a maximum is reached, but the decrease at lower depths is more gradual. The moisture equivalent of a particular soil is not markedly affected by changes in carbonate content. The colour of the soil and the proportion of carbonate present were closely related. A. G. POLLARD.

Soil studies at the Missouri station. M. F. MILLER, R. BRADFIELD, and F. L. DUDLEY (Missouri Agric. Exp. Sta. Bull., 1925, [228], 77—84; Chem. Abstr., 1926, **20**, 2218).—Neutral or acid salts are much more efficient flocculating agents than alkaline salts. Treatments which lowered the Sörensen value of the clay caused liberation of more equivalents of total bases than were absorbed; those isohydric with the clay caused equivalent exchange, and those raising the p_H value of the clay caused the absorption of more equivalents of bases than were liberated. Experimental evidence indicates that the good effects on crops of liming are largely due to the soluble calcium available to the plant rather than to the neutralisation of acidity. A. A. ELDRIDGE.

Soil studies at the Wisconsin Experiment Station. ANON. (Wisconsin Exp. Sta. Bull., 1925, [373], 41—51; Chem. Abstr., 1926, **20**, 2218).—The decomposition products of decaying manure assist in rendering the soil potash-soluble. To obtain the maximum atmospheric nitrogen fixation from lucerne, more phosphate is required than most soils contain. An appreciable amount of aluminium is present in the soil solution only when it is strongly acid or alkaline; the amount could be determined from the reaction. Aluminium ions are no more toxic than the corresponding amount of hydrogen ions. With barley, maize, and soya beans, part of the favourable effect of liming acid soils was due to the precipitation of aluminium. A. A. ELDRIDGE.

Soil and plant relationships. M. M. MCCOOL and J. D. ROMAINE (Soil Sci., 1926, **22**, 31—34).—The heat of wetting of a number of plant materials (seeds, leaves, roots) was determined, and found to vary with the nature of the plant, season, and previous fertiliser treatment of the soil. The possibility and value of correlating the heat of wetting of plants with soil fertility are indicated. A. G. POLLARD.

Sweet clover in relation to the accumulation, loss, and conservation of nitrates in soil. A. L. WHITING and T. E. RICHMOND (Soil Sci., 1926, **22**, 1—19).—The value of sweet clover as a green manuring crop is demonstrated. Rapid nitrification follows the ploughing-in of the green crop, and ample nitrate can thus be accumulated for a subsequent grain crop. Used as a catch crop, sweet clover conserves much soil nitrate as organic nitrogen, and losses by leaching during rainy seasons are thereby considerably reduced. A. G. POLLARD.

Influence of form, soil zone, and fineness of lime and magnesia incorporations upon the outgo of sulphates and nitrates. W. H. MACINTIRE (Soil Sci., 1926, **22**, 21—30; cf. B., 1926, 640).—The rate of leaching out of nitrates and sulphates from soils is used as a measure of increased biological activity following the application of lime in various forms. The records of a four-year period are discussed. A. G. POLLARD.

Residual effects of forty years continuous manurial treatment. III. Ultimate fate and some physical and chemical effects of applied lime. J. W. White and F. J. Holben (Soil Sci., 1926, 22, 61—74).—The rate of decomposition of limestone in field soils follows closely the changes in soil reaction. In the soil studied applications of lime maintained a strongly alkaline reaction. There was, however, a considerably increased proportion of soluble organic matter and nitrate in the limed plots. During the growing season lime tended to reduce the proportion of water-soluble potassium in the soil. The percentage of carbon and nitrogen was greater in limed plots. No measurable effect of lime on the moisture-holding capacity of soil was observed, but the plough draft was markedly reduced.
A. G. Pollard.

Homogeneous carbon disulphide emulsion. W. E. Fleming (J. Agric. Res., 1926, 33, 17—20).—See B., 1925, 940.

Humus in sewage sludge. Bach.—See XXIII.

Patents.

Fertiliser. F. Tharaldsen and E. Lie (Nor. P. 39,805, 22.1.23).—Crude calcium cyanamide is treated with sufficient phosphoric acid to convert the calcium oxide present which is not combined as cyanamide, into soluble calcium phosphate. C. T. Gimingham.

Treatment of acid-soluble minerals containing potassium. Norsk Hydro-Elektrisk-Kvaelstof-Aktieselskab (Nor. P. 40,027, 29.5.23).—The finely-ground mineral, *e.g.*, leucite, is treated with a solution of a salt of urea, preferably the phosphate. A product is obtained, containing potassium in a soluble form, which, on drying, may be used directly as a fertiliser.
C. T. Gimingham.

XVII.—SUGARS; STARCHES; GUMS.

Patents.

Separating and collecting organic acids and bases from beetroot molasses. Y. Takayama (U.S.P. 1,595,529, 10.8.26. Appl., 30.6.24).—See E.P. 233,196; B., 1925, 568.

Centrifugal machines (E.P. 252,339).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Rôle of proteolytic enzymes in decomposition of the herring. Almy.—See XIX.

Patent.

Manufacture of non-alcoholic beer etc. J. F. Meyer (U.S.P. 1,593,191, 20.7.26. Appl., 2.6.25. Conv., 9.3.21).—A cereal is malted for 7 or 8 days at about 19° and mashed for about 10 hrs., at 10°, to produce an acid mash. The malt wort is extracted and subjected to a short alcoholic fermentation to produce sufficient acid-reacting products to modify the sugar contents as to flavour. The fermentation is then stopped by cooling and the product is clarified out of contact with air.
H. Holmes.

XIX.—FOODS.

Sweetened condensed milk. IV. Refractometric method for determining total solids. F. E. Rice and J. Miscall (J. Dairy Sci., 1926, 9, 140—152; Chem. Abstr., 1926, 20, 2211).—Formulæ and tables are derived for the determination of total solids in sweetened condensed milk (*a*) containing 8% of fat, and (*b*) skim. The method is trustworthy when the sucrose content varies between wide limits provided the fat content approximates to that for which the formula has been prepared. A. A. Eldridge.

Colour reactions of vitamin-A. T. T. Cocking and E. A. Price (Pharm. J., 1926, 117, 175—178).—Of the various colour reactions and modifications for the estimation of vitamin-*A* in cod-liver oil, the most satisfactory was found to be that given by a solution of antimony trichloride in chloroform. The blue coloration given by a solution of trichloroacetic acid in chloroform was found to be caused by the presence of traces of phosgene in the commercial acid. The colour of the oil and the amount of unsaponifiable matter are useless as criteria of vitamin content. The vitamin activity of cod-liver oils may vary as much as 8 : 1, Newfoundland oils usually having a much greater activity than Norwegian oils. Details of a method of estimating vitamin activity using a solution of antimony trichloride in chloroform in conjunction with a Lovibond tintometer are given (cf. Carr and Price, A., 1926, 870). Special precautions are not necessary as the reagent is not affected by traces of moisture, but the presence of carrotene vitiates the results. C. O. Harvey.

Sweet potato starch in cornflour and arrowroot. J. R. Stubbs (Analyst, 1926, 51, 400—402).—The granules of sweet potato starch are very varied in shape and size and often have a hilum. They resemble a mixture of tapioca, sago, maize, and rice starches, whilst some have "facets." Sweet potato starch was recently found in a sample submitted as arrowroot, and during the last seven years one sample of cornflour was found to consist entirely of sweet potato starch, whilst three samples of arrowroot contained sweet potato starch in conjunction with maranta starch. D. G. Hewer.

Rôle of the proteolytic enzymes in the decomposition of the herring. L. H. Almy (J. Amer. Chem. Soc., 1926, 48, 2136—2146).—The rapid decomposition of immature herrings (used in sardine packing) which, when caught, have food in their stomachs, is due to the softening of the abdominal wall brought about by the trypsin of the pyloric ceca, which is more active and present in greater amount when food is present. This allows the flesh to be attacked by bacteria. The pepsin extracted from the stomach of the fish is most active at 37° and at p_H 2·5—2·85, and is present in greater amount in the stomachs of feeding fish. The trypsin from

the pyloric ceca is most active at blood heat, and at p_H 8·5—9·5, but it acts slowly at p_H 6·85.

F. G. Willson.

"Blowing" of canned fruit due to chemical action. G. W. Monier-Williams (Analyst, 1926, **51**, 402—403).—The gas from five blown tins of loganberries with sterile contents was found to consist chiefly of hydrogen. The lacquer on the inner surface of the cans had given way in several longitudinal streaks, and there the tin had been dissolved, leaving the iron exposed. This latter was extensively corroded, particularly along the edges of the seam, presumably by the fruit acids. The total quantities of tin (*a*) and iron (*b*) in the contents of the tins, which averaged 550 g., were (*a*) 16—20 mg. in four cases and 66 mg. in one, and (*b*) 80—175 mg.

D. G. Hewer.

Freshly ground coffee and "blown" tins. J. W. Black (Analyst, 1926, **51**, 403—404).—A quantity of Costa Rica coffee was ground after keeping for 8 days from the roasting time, and the evolution of gas immediately determined. For 200 g. of coffee, 52 c.c. of gas were collected in 1 hr., 90 c.c. in 5 hrs., and 132 c.c. in 48 hrs., and this result is regarded as typical.

D. G. Hewer.

See also A., Sept., 903, **Microscopical study of freezing of gel** (Hardy). 904, **Freezing of gelatin gel** (Moran). 905, **Colloid chemistry of rennin coagulation** (Palmer and Richardson).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Use of the potentiometer in the quantitative analysis of alkaloidal solutions. J. C. Krantz, jun. (J. Amer. Pharm. Assoc., 1925, **14**, 294—299; Chem. Abstr., 1926, **20**, 2391).—Since the molarity of an alkaloidal salt solution does not affect the p_H, a definite excess of acid added to variable amounts of alkaloidal salts and diluted to a definite volume at which the ionisation of the acid is practically complete will yield solutions of which the p_H is a function of the excess of acid present. Experimental results are interpreted by means of a curve constructed, *e.g.*, for quinine, strychnine, or atropine in 0·1*N*-hydrochloric acid. The method is valueless for caffeine.

A. A. Eldridge.

Dissociation and volumetric determination of the cinchona alkaloids. C. Morton (Pharm. J., 1926, **117**, 168—173).—The equilibria in solutions of diacid bases and of their hydrochlorides are considered, and a determination of the basic constants of the chief cinchona alkaloids from electrometric measurements of the hydrion concentrations of solutions of the hydrochlorides is described. Though a mixture of piperidine and quinoline can be accurately titrated to the half-neutralisation point with a wide choice of indicators, in the case of quinine bromocresol-purple is the only suitable indicator, and the titration error is about 10% unless a colour standard is used. The optimum titration conditions for the cinchona alkaloids in 0·05*N*-solutions are given in tabular form.

C. O. Harvey.

Determination of morphine in poppy extracts. C. T. Bennett and D. C. Garratt (Pharm. J., 1926, **117**, 149).—Poppy extracts, when mixed with lime and water as in the B.P. method for the determination of morphine in opium, yield a difficultly filterable mixture, and the nature of the filtrate prevents the clean separation of morphine. Extraction with a mixture of cresol and amyl alcohol gave concordant results but was tedious. Alcohol (90%) and *iso*propyl alcohol were found to be satisfactory solvents, and methods based on the use of the latter are described, the morphine in the extract being determined by modifications of the B.P. process, using methyl-red as indicator in place of methyl-orange.

C. O. Harvey.

Assay of extract of aconite. C. W. Cornwell and A. J. Jones (Pharm. J., 1926, **117**, 197—199).—In assaying extract of aconite the extraction of the ether-soluble alkaloids in the dry material does not yield results identical with those obtained by shaking out the alkaline aqueous solution with ether, especially when the alkaloids have undergone degradation. Alcohol must be excluded from the assay process. The distribution ratio for crystalline aconitine between water and ether is practically 1 : 11, and the solubility of the alkaloid in ether (given in the U.S.P. as 1 in 65) is practically confirmed under assay conditions. The alkaloidal residue is easily decomposed.

C. O. Harvey.

Assay of belladonna leaves. C. M. Caines and N. Evers (Pharm. J., 1926, **117**, 179—180).—The various methods in use for the assay of belladonna leaves are discussed and criticised, comparative figures being given, and it is concluded that the official B.P. method is the best. Methyl-red is recommended as the most satisfactory indicator for use in the volumetric method.

C. O. Harvey.

Detection and determination of glycerol in tobacco. A. C. Chapman (Analyst, 1926, **51**, 382—386).—10 g. of the sample and a few grams of anhydrous sodium sulphate are extracted in a Soxhlet thimble with pure acetone for one day, the thimble left full overnight, and extraction continued for a few hours the next day. The solvent is evaporated, the residue dissolved as far as possible in 20 c.c. of warm alcohol, and water (about 150 c.c.) added until precipitation of the resins appears complete. These are extracted by shaking with petroleum spirit, after which the aqueous layer is run off, the spirit and funnel washed with water, and the main solution and washings are gently distilled. When the volume is reduced to about 30 c.c. the cooled liquid is filtered into a weighed carbonic acid flask. The distillate is evaporated to about 5—10 c.c. and added to the residue, and the whole evaporated to a syrup at about 80° in a current of air until a marked odour of nicotine is apparent. A portion of the crude extract is weighed on a piece of glass into a long-necked flask of the type used in Perkin's modified Zeisel apparatus.

A flask containing a suspension of red phosphorus in water is attached to the long-necked flask, and immersed in a water bath at 60—70°; this serves to retain any iodine liberated. It is connected with a flask containing alcoholic silver nitrate and a guard flask to retain the *iso*propyl iodide formed. 15 c.c. of hydriodic acid (d 1·71) are placed in the long-necked flask, which is heated in an oil bath at 135—140°, and a slow current of carbon dioxide passed through. If an appreciable amount of glycerol is present precipitation of silver iodide begins in 20 min. and, after complete precipitation, the salt is separated and weighed, and the weight multiplied by 0·392 gives the equivalent of glycerol. For the detection of glycerol, after extraction with acetone for some hours, and removal of resins, an aqueous extract of small bulk is prepared, and traces of water are removed under reduced pressure. The flask is then connected to a small Bruhl apparatus and the distillate boiling at 120—130° at 5 mm. pressure is dissolved in water, the greater part of the nicotine removed with ether, and the remainder of the base precipitated with silicotungstic acid, filtered off, and the filtrate evaporated to small bulk after neutralisation with soda. The water is evaporated, the residue distilled under 2—3 mm. pressure, and the distillate identified as glycerol. Control experiments with glycerol-free tobacco showed the very gradual formation of a very small amount of silver iodide precipitate, and a deduction of 0·06 g. from the weight of silver iodide, corresponding with 10 g. of glycerol-containing tobacco, should be made. The results were accurate to within 0·1% of the glycerol added.

D. G. HEWER.

Chemical constituents of the oleo-resin and fatty matter of Indian valerian root. K. BULLOCK (Pharm. J., 1926, **117**, 152—157; cf. B., 1925, 825).—On steam distillation valerian root yielded 0·35% of oil (n 1·5025, d^{19} 0·9819), consisting principally of a sesquiterpene hydrocarbon plus small quantities of a sesquiterpene alcohol and valeric and formic acids in the combined state. Saturated acids (probably a mixture of stearic and palmitic acids) occur in the free condition, as also do a small quantity of unsaturated acids of the oleic series and a notable quantity of valeric acid. A petroleum spirit extract of the root was found to contain (in addition to the constituents of the steam-distilled oil) arachidic acid, hentriacontane, neutral and acidic resinous material, with some linoleic and linolenic acids. Terpenes or borneol were not found in any of the material examined.

C. O. HARVEY.

Automatic continuous percolator [for extraction of drugs]. D. S. RATTRAY (Pharm. J., 1926, **117**, 195—196).—The inner tube of a Greiner and Friedrich extraction-tube (having its drug-containing vase removed) is lengthened so that it projects below the outer tube, and both tubes dip below the surface of the extracting liquid in the container. The apparatus is connected to a filter pump via a condenser and water trap, and by carefully adjusting the pump a continuous, steady flow of the liquid through the drug in the extraction tube is maintained.

C. O. HARVEY.

Differentiation of veronal, proponal, and luminal. L. EKKERT (Pharm. Zentr., 1926, **67**, 481—482).—If the substances be dropped on to molten sodium hydroxide, veronal gives an acid, rancid odour; luminal gives first a pleasant odour, which becomes piercing, the veronal becoming yellow to yellowish-red; proponal gives first a spicy odour, which becomes piercing. Any of the substances dissolved with salicylaldehyde in alcohol and covered with a layer of strong sulphuric acid gives a red coloration at the zone of contact. If luminal, even in traces, be treated with formaldehyde and strong sulphuric acid, it gives a red coloration, gradually at room temperature and in 1 min. on the boiling water-bath; veronal and proponal give only a yellowish coloration even after some minutes on the water-bath. Known tests are reviewed.

B. FULLMAN.

Volatility of benzoic acid. A. F. LERRIGO (Analyst, 1926, **51**, 405—406).—Benzoic acid crystals prepared by dissolving 0·5 g. of pure acid in dry methylated ether and in ether saturated with water, spontaneously evaporating the ether, and exposing for 24 hrs. to room temperature, were heated in the oven for observed periods at different temperatures. Smaller and finer crystals were obtained from the dry ether, and the rate of loss increased but slowly up to 50°, being about 0·001 g. per hr. at 40°, but at 70° the loss was more than 0·02 g. per hr.

D. G. HEWER.

Reaction between lead subacetate and phenol. G. A. MEDLEY (Pharm J., 1926, **117**, 149—150).—As the precipitate formed by adding basic lead acetate to aqueous solutions of phenol is insoluble in phenol, the conclusion is drawn that its formula is $(C_6H_5O_2)Pb$ and not $C_6H_5O.Pb.OH$. The absence of a precipitate when normal lead acetate is used is probably due to the acidity developed in normal acetate solutions; precipitation with the basic acetate is prevented by adding a few drops of dilute acetic acid. Other phenols give similar precipitates.

C. O. HARVEY.

Micrographic test for tartaric acid in solutions containing it. M. FRANÇOIS and C. LORMAND (J. Pharm. Chim., 1926, [viii], 4, 54—61).—The presence of d-tartaric acid may be shown by adding calcium acetate, the resulting crystals of calcium d-tartrate being recognisable microscopically. These crystals form in a solution of d-tartaric acid containing more than 0·15 g. of the acid per litre and less than 1 g. of citric acid per litre if the latter be present. The presence of tartaric acid may be confirmed by testing the precipitate with resorcinol and sulphuric acid.

B. FULLMAN.

Chaulmoogra oil and its saponification. A. J. GELARIE and F. R. GREENBAUM (Amer. J. Pharm., 1926, **98**, 411—414).—If treated with the quantity of sodium hydroxide indicated by its saponification value, chaulmoogra oil is incompletely saponified. Saponification is effected by shaking 50 g. of the oil with 14·25 g. of sodium hydroxide in 50 c.c. of water for ½ hr., and leaving the mixture for at least 12 hrs. in the cold. The resulting soap shows 0·7—0·8% of free alkalinity, and

yields a 10% solution. The oil is saponified in ½ hr. if 13 c.c. be shaken with 40 c.c. of 10% alcoholic sodium hydroxide. The soap obtained has alkalinity 1·4%, and yields a 40% solution in the cold. The solution is freed from alcohol by distillation *in vacuo* at 40—50°, when it congeals; it re-liquefies when the alcohol is replaced by an equal quantity of water, the free alkalinity being then 0·7%. This solution is neutralised with concentrated hydrochloric acid and, on addition of water till the sodium chloride concentration is 0·8%, has a soap concentration equivalent to 7—10% of oil. This solution may be used intramuscularly or intravenously.

B. Fullman.

Analysis and composition of commercial glycerophosphates. G. J. W. Ferrey (Pharm. J., 1926, **117**, 159—162).—Esterification of phosphoric acid by glycerol at 105—110° yields chiefly the dibasic mono-ester $(OH)_2C_3H_5\cdot O\cdot PO(OH)_2$, but at temperatures above 110° condensation occurs to the di-ester which, being monobasic, causes lowering of the proportion of metallic radical in the salts. As normal sodium glycerophosphate is slightly alkaline to phenolphthalein, in order to determine exactly the alkaline or acid impurity a more satisfactory indicator is thymolphthalein, the p_H values of neutral glycerophosphate solutions being found to be within the range 8·5—9·5. After neutralisation to thymolphthalein, the titration of the glycerophosphate is carried out with dimethylaminoazobenzene as indicator. Titration with acid and alkali gives trustworthy results, but only in the absence of organic acids and inorganic phosphates. The products of most English manufacturers were found to be reasonably pure and free from di-ester, but considerable variations in the amounts of water of crystallisation were observed, particularly with the magnesium and the 50% sodium salts.

C. O. Harvey.

Carbon tetrachloride in pharmacy. G. E. Trease and H. Tingey (Pharm. J., 1926, **117**, 150—152).—As a solvent for the preparation of certain oleo-resins carbon tetrachloride has no advantages other than that of its non-inflammability, and it appears to have little use as an extracting medium for alkaloidal drugs. Carbon tetrachloride has an advantage over chloroform and iodoform as a colour reagent for phenols in that it gives characteristic reactions with a number of *o*- and *m*-substituted phenols, but not with the *p*-substituted phenols examined. The colours are probably due to dyes of the aurin type.

C. O. Harvey.

Utilisation of ethylene obtained by cracking of petroleum for the production of alcohol. W. Gerr and S. Popov (Neftjanoe Chozjajstvo, 1926, **10**, 88—82; Chem. Zentr., 1926, II., 667).—Gas obtained from solar oil by cracking consisted of 54% of methane and its homologues, 12·2% of hydrogen, and 33·8% of unsaturated hydrocarbons. It was passed over calcium chloride, over wood charcoal to remove higher homologues of ethylene, and through an absorption tube kept at 40° and containing glass beads or glass wool, in which a stream of a 1% solution of silver sulphate in 94% sulphuric acid absorbed the ethylene. The ethylsulphuric acid obtained was decomposed with water and the alcohol distilled off, 33·82 g. of alcohol being obtained from 300 litres of gas. When the gas has not been previously freed from the higher olefines the yield of alcohol is practically the same, and an oil insoluble in water is also obtained. The gas freed from ethylene burns without smoke.

H. Moore.

Determination of arsenic in chemicals by the electrolytic method. N. Evers (Pharm. J., 1926, **117**, 183—184).—A modified apparatus for the electrolytic determination of arsenic is described, the method of preparing and connecting the cathode being simplified. A list of chemicals, mostly pharmaceutical products, which may be directly tested is given, and special methods of preliminary treatment are described.

C. O. Harvey.

Genus *Mentha*. VI. Volatile oil of a strain of Japanese peppermint grown at Madison, Wisconsin. G. C. Jenison and R. E. Kremers (J. Amer. Pharm. Assoc., 1925, **14**, 495—498; Chem. Abstr., 1926, **20**, 2392).—A strain of Japanese peppermint, the oil of which is normally rich in *l*-menthol, produced an oil containing 85% of pulegone and a small quantity of *l*-limonene; neither menthol nor menthone could be found.

A. A. Eldridge.

Obtaining camomile oil. E. Kárpáti (Riechstoffind., 1926, 37—39; Chem. Zentr., 1926, II, 660).—An intimate mixture of 3 pts. of camomile flowers and 2 pts. of residues is distilled with steam at a pressure of 7—8 atm. The average oil content of Hungarian camomile is 0·318%.

B. Fullman.

Essential orange-flower extract oil. Elze (Riechstoffind., 1926, 29—30; Chem. Zentr., 1926, II, 660).—On extraction with light petroleum, 1000 kg. of fresh orange flowers gave a product which yielded on treatment with alcohol and distillation 0·165 kg. of oil (d 0·892, α_D − 5°, linalyl acetate 47·04%) containing a number of terpenes, methyl anthranilate, etc. The residues of distillation contain 0·07 kg. of oil (d 1·0049, α_D + 1·5°, 41·2% of linalyl acetate). In the residues of distillation of both oils farnesol occurred.

B. Fullman.

Essential oil from the flowerheads of *Perovskia atriplicifolia*, Benth. M. G. Rao (J. Indian Chem. Soc., 1926, **3**, 141—147).—The flower heads yield 1% of a light green oil having d^{30}_{30} 0·8943, n_D^{30} 1·4748, α_D^{30} + 8·53°, acid value 0·2, ester value 30·4 (after acetylation 49·22). The oil contains about 50% of terpenes, chiefly *d*-α-pinene, β-pinene, and camphene; 15—18% of alcohols and esters (mainly *d*-borneol and its acetate); and sesquiterpenes, α-caryophyllene and aromadendrene having been identified. The oil may prove to be a valuable source of *d*-borneol.

G. M. Bennett.

Essential oil of *Boronia citriodora* and the occurrence of citronellol. A. R. Penfold (J. Proc. Roy. Soc. N.S.W., 1925, **59**, 35—40).—Steam distillation of the

leaves and terminal branchlets of this Tasmanian alpine plant yields 0·71—0·93% of an oil (d^{15}_{15} 0·8814—0·8822, $\alpha - 2·8°$ to $- 3·8°$, n_{20} 1·4608—1·4611, ester value 42·10—69·88, ester value after acetylation 239·46—241·24, acid value 4—5, solubility in 70% alcohol 1 : 1), containing citronellol (80·38—82·31%), citronellol esters, principally the acetate, with some valerate, *d*-α-pinene, sesquiterpene, a paraffin (m.p. 64—65°), and small quantities of a phenolic substance and free capric acid. Citral, citronellal, and geraniol are absent. E. H. SHARPLES.

Essential oils from the leaves of *Murraya Koenigii* (Spreng.), *Murrayaexotica* (Linn.), and *M. exotica* var. *ovatifoliolata* (Engler). A. R. PENFOLD and J. L. SIMONSEN (J. Proc. Roy. Soc. N.S.W., 1925, **59**, 146—155).—In each case the leaves were steam-distilled. *M. Koenigii* from Dehra Dun yields 0·04% of a pale yellow oil (d^{30}_{30} 0·8711, n^{30} 1·478, $\alpha - 18·2°$, acid value 1·1, saponif. value 11·06, saponif. value after acetylation 31·83), containing α-pinene, *l*-caryophyllene, esters of palmitic acid, and *dl*-sabinene. On oxidation with alkaline potassium permanganate, *dl*-sabinene yields dl-*sabinenic acid* (m.p. 84—85° anhyd.). *M. exotica* from Dehra Dun yields 0·01% of a dark, unpleasant smelling oil (d^{15}_{15} 0·9023, n_D^{22} 1·496, saponif. value 8·87, saponif. value after acetylation 72·53, insoluble in 10 vols. of 80% alcohol), containing *l*-cadinene, a second sesquiterpene, and possibly methyl anthranilate. *M. exotica* var. *ovatifoliolata* from Queensland yields 0·06—0·15% of oil (d^{15}_{15} 0·9117—0·9126, n_D^{20} 1·499—1·5008, $[\alpha]_D - 10°$ to $- 24·7°$, saponif. value 19·37—26·18, saponif. value after acetylation 68·55), containing principally bisabolene and an unidentified sesquiterpene with small amounts of a sesquiterpene alcohol, free palmitic acid, and esters of caprylic acid. E. H. SHARPLES.

Essential oil of *Eriostemon myoporoides* (de Candolle). A. R. PENFOLD (J. Proc. Roy. Soc. N.S.W., 1926, **59**, 206—211).—Steam distillation of the leaves and terminal branchlets yields 0·71—0·77% of a mobile, bright yellow oil having d^{15}_{15} 0·8580—0·8789, $\alpha + 29·2°$ to $+ 36·75°$, n^{20} 1·4687—1·4740, saponif. value 4·61—6·38, saponif. value after acetylation 14·59—24·69. It is insoluble in 12 vols. of 80% alcohol (by wt.) and contains *d*-α-pinene (75—85%), ocimene, a sesquiterpene, ledum camphor, methyl anthranilate, a paraffin of m.p. 64—65°, and traces of a phenolic substance. E. H. SHARPLES.

Essential oils of *Melaleuca linariifolia* (Smith) and *M. alternifolia* (Cheel). A. R. PENFOLD (J. Proc. Roy. Soc. N.S.W., 1926, **59**, 306—324).—The leaves and terminal branchlets were distilled in both cases. *M. linariifolia* gives an average yield of 1·5% of oil having d^{15}_{15} 0·8927—0·8992, $\alpha + 3·3°$ to $+ 6·8°$, n^{20} 1·4752—1·4780, ester value 1·3—2·67, ester value after acetylation 58·23—82·10. *M. alternifolia* yields 1·76—1·83% of oil having d^{15}_{15} 0·8958—0·8961, $\alpha + 6·8°$ to 7·4°, n^{20} 1·4782—1·490, ester value 3·67—7·35, ester value after acetylation 79·36—83·64. The principal constituents of both oils are α- and γ-terpinene, cymene, cineole (16—20% in the former, 6—8% in the latter), Δ^1-terpinenol-4 (naphthylurethane, m.p. 104—105°; nitrosochloride, m.p. 115—116°), sesquiterpenes, chiefly cadinene and the corresponding sesquiterpene alcohol. Small amounts of *d*-α-pinene and a phenol are present. *M. linariifolia* contains an unidentified constituent possessing the physical properties of sabinene. E. H. SHARPLES.

Essential oil of *Baeckea Gunniana*, var. *latifolia* (F.v.M.). A. R. PENFOLD (J. Proc. Roy. Soc. N.S.W., 1926, **59**, 351—355).—Steam distillation of the leaves and terminal branchlets yields 0·33—0·74% of a dark brown product (m.p. 43—47°, d^{15}_{15} 0·9578—d^{26}_{15} 0·9550, $\alpha + 15·6°$ to $+ 23·0°$, n^{20} 1·5038—1·5059, ester value 11·44—23·19, ester value after acetylation 138·38—151·80, solubility in 70% alcohol (by wt.) 1 : 2—3), containing eudesmol (60%), eudesmene, β-pinene, and a small quantity of an unidentified phenol and valeric acid ester, and a yellow stearoptene, $C_{13}H_{18}O_4$ (m.p. 103·5—104°). E. H. SHARPLES.

Germicidal values of some Australian essential oils and their pure constituents, together with those for some essential oil isolates and synthetics. III. A. R. PENFOLD and R. GRANT (J. Proc. Roy. Soc. N.S.W., 1926, **59**, 346—350; cf. B., 1925, 334).—Rideal–Walker coefficients are given. The influence of the degree of dispersion on the magnitude of the coefficient is discussed. For the rapid detection of small quantities of cymene in admixture with terpenes the determination of the Rideal–Walker coefficient is of considerable value, its presence raising the number out of all proportion to the percentage present. The term "biometric test" is applied to this form of analysis. E. H. SHARPLES.

Polarimetric examination of cade oils. R. MASSY (J. Pharm. Chim., 1926, [viii], **4**, 61—65).—Polarimetric examination of oils obtained (presumably by treatment with alkali and steam distillation) from the tars yielded on carbonisation in a closed vessel of several coniferous woods—*Juniperus oxycedrus*, *J. phœnicea*, *J. thurifera*, and *Pinus halapensis*—indicated that those from the trunk of *J. oxycedrus*, from the root, trunk, and branches of *J. phœnicea*, and from the root and trunk of *J. thurifera* were lævorotatory, the other root, branch, and trunk oils being slightly dextrorotatory or almost inactive. The lævorotatory nature of the product from true cade oil (from *J. oxycedrus*) does not therefore characterise it, as indicated by Huerre (J. Pharm. Chim., 1926, [viii], **3**, 314). Only the oil derived from *Cedrus atlantica* was strongly dextrorotatory. B. FULLMAN.

Testing of cade oil. R. HUERRE (J. Pharm. Chim., 1926, [viii], **4**, 65—66).—Polemical. A reply to Massy (cf. preceding abstract). B. FULLMAN.

See also A., Sept., 902, **Alkalinised solutions of salvarsan** (HUNTER and PATRICK). 917, **Reduction of carbon monoxide** (ELVINS and NASH). 918, **Catalytic dehydration of methyl alcohol and properties of hydrous aluminium oxide catalyst** (HOWARD); **Catalytic actions of various types of reduced copper on alcohols** (HARA). 936, **Synthesis and hydrolysis**

of a glycerolmonophosphoric diester (Bailly and Gaumé).

Patents.

Manufacture of new complex antimony compounds. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 247,986, 19.2.26).—Antimony pentoxide or pentachloride reacts with a solution of thioglycollic acid to produce an acid, $Sb(S\cdot CH_2\cdot CO_2H)_5$, the easily soluble salts of which can be isolated by neutralising the acid solution with alkalis or alkaline-earths and evaporating. On acidifying the solutions of these salts, or on evaporating the original reaction product directly, thioglycollic acid is partially split off and another new *acid* separates, containing 40% Sb as against 20·8% for the pentathioglycollic acid. The second acid also yields water-soluble salts. Both series of salts have specific pharmaceutical properties. A. Davidson.

Preparation of perfumes. I. G. Farbenind. A.-G., Assees. of A. Eisenhut (G.P. 428,548, 12.9.24).—The fraction boiling above 150° of the product, rich in alcohols, obtained by reducing carbon monoxide under pressure, is hydrolysed; or the reduction product, especially the fraction boiling at 150—200°, is esterified with aromatic acids. B. Fullman.

Process for extracting the organs of animals, plants, etc. Maschinebau-Anstalt Humboldt (G.P. 430,087, 22.7.24).—The material is brought into the disperse condition by means of rapidly running mills, a small quantity of a solvent or an indifferent liquid being added at the start, and the dispersion is extracted in the usual way. The process is applicable to the extraction of cinchona bark, wormwood, *nux vomica*, liver, spleen, dye and tanning woods, crude anthracene, etc. W. G. Carey.

Pharmaceutical product. [α-Hydroxylepidine dimethylaminoethyl ether.] J. Callsen, Assr. to Winthrop Chemical Co. (Reissue 16,394, 27.7.26, of U.S.P. 1,572,768, 9.2.26. Appl., 18.6.26).—See B., 1926, 464.

Producing a substance adapted to tampon wounds. R. Vogel (U.S.P. 1,593,814, 27.7.26. Appl., 13.2.25. Conv., 19.2.24).—See E.P. 229,644; B., 1925, 970.

Manufacture of [organic] sulphocyano-derivatives. A.-G. für Anilin-Fabr., Assees. of O. Spengler and W. Müller (U.S.P. 1,594,697, 3.8.26. Appl., 2.9.25. Conv., 24.9.24).—See E.P. 240,420; B., 1925, 1012.

Preparation of a solution of cyanamide from commercial calcium cyanamide. J. Breslauer, Assr. to Comp. de l'Azote et des Fertilisants S.A. (U.S.P. 1,595,754, 10.8.26. Appl., 14.11.24. Conv., 20.11.23).—See E.P. 225,206; B., 1925, 785.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Developer containing diaminophenol and p-phenylenediamine in bisulphite solution. L. Lobel and L. J. Bunel (Bull. Soc. franç. Phot., 1926, [iii], **13**, 143—147).—The following developer was studied: water, 1 litre, sodium bisulphite solution (*d* 1·32), 75 c.c., *p*-phenylenediamine base, 13 g., diaminophenol hydrochloride, 5 g. This mixture gives a developer of normal rapidity, whilst diaminophenol does not develop in bisulphite solution, and *p*-phenylenediamine develops only in sulphite solution and then very slowly. The high development rate of the mixture is due to the formation of sodium sulphite and *p*-phenylenediamine sulphite by interaction of the bisulphite with the *p*-phenylenediamine. The mixture is far more stable in air than amidol developer; the stability approaches that of metol–quinol. It is very sensitive to the presence of bromide ions, whereas diaminophenol is scarcely affected by bromide. W. Clark.

See also A., Sept., 920, **Influence of adrenaline on the photographic plate** (Vollmer). 923, **Colloidal aurous oxide. Gold toning of photographic papers.** (Steigmann).

Dyeing processes using Indigosol O. Friedländer.—See VI.

Patents.

Changing the light-sensitiveness of photographic emulsions. S. E. Sheppard, Assr. to Eastman Kodak Co. (U.S.P. 1, 591, 499, 6.7.26. Appl., 2.12.25).—A silver halide photographic emulsion is incorporated, at any stage of its preparation, with an organic compound in which a sensitising substance is combined with a group which renders it latent. Subsequently, by addition of a suitable enzyme the sensitiser is released in an active form. For example, a mustard oil sensitiser is prepared by treating the seeds of black mustard with boiling alcohol, extracting the residue with cold water, evaporating the solution to a small volume, acidifying with tartaric acid, and again extracting with alcohol. The syrup obtained, after being neutralised with potassium carbonate, constitutes a latent sensitiser, the sensitising substance, allyl mustard oil, being combined with a monose radical. The syrup is mixed with the silver halide emulsion and the active sensitising agent liberated by addition of an enzyme prepared from mustard seed. R. B. Clarke.

Preparation of photographic developers. P. Schestakoff and B. Merejkovsky (E.P. 255,925, 24.4.25).—See F.P. 600,532; B., 1926, 566.

Photographic layer. R. Schwarz (U.S.P. 1,594,470, 3.8.26. Appl., 10.9.25. Conv., 12.9.24).—See G.P. 413,217; B., 1925, 787.

XXII.—EXPLOSIVES; MATCHES.

Chemical stability of nitroglycerin powders. M. Tonegutti (Z. ges. Schiess- u. Sprengstoffw., 1926, **21**, 127—128).—In the Abel heat test, acid ballistites give the same results as normal ballistites, whereas according to the Taliani and silvered vessel tests the stability of the former is much less. Cordite M.D. and the Austrian powders M/97 and M/97A show the reverse behaviour,

as acid samples of these have a low stability in the Abel test, whilst by the other methods little difference is found between acid and normal samples. This behaviour of acid cordites is attributed to the vaseline present in them. The low stability of acid cordites in the Abel test is attributed to the presence of traces of acetone, which causes the lowering of the heat test value when the powders become acid. S. BINNING.

Nitrous esters of cellulose. F. BLECHTA (Z. ges. Schiess- u. Sprengstoffw., 1926, **21**, 39—41).—Methods used by previous workers when investigating the possibility of producing nitrous esters of cellulose are criticised. By using Meisenheimer's method for determining nitrites in presence of nitrates it is shown that by the action of nitrous acid on cellulose nitrous esters are formed to an extent that increases with the amount of nitrous acid in the nitric acid used. Eventually, however, with a 30% content of nitrous acid, complete solution of the cellulose residue takes place so that it is impossible to produce nitrocelluloses with any desired content of nitrous ester. In the ordinary process of nitration, nitrous esters are not formed, and even if they were formed owing to abnormal conditions, they would not affect the stability of the boiled nitrocellulose as they would be destroyed during its stabilisation.
S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Corrosion of iron water mains. C. F. HICKETHIER (Rev. Fac. Cien. Quím., 1925, **3**, 73—92; cf. B., 1925, 333).—A discussion of the factors affecting the corrosion of iron water mains and of protective measures, with special reference to the water supply of Buenos Aires.
G. W. ROBINSON.

Modification of the Kjeldahl method for determining organic nitrogen in sewage effluents etc. J. W. H. JOHNSON (Analyst, 1926, **51**, 405).—Large amounts of free and saline ammonia are first distilled off, as usual, followed by distillation of nitrites after acidification with 1 in 4 sulphuric acid. Nitrates are then reduced by addition of 0·5 g. of zinc dust in the form of an ammonia-free suspension and 10 c.c. of sulphuric acid, and the solution is refluxed for 15 min. so that no loss in the intermediate nitrous stage occurs. The process is then completed as usual. D. G. HEWER.

Anaerobic decomposition of sewage sludge. K. N. KOROLKOV (Trav. Comm. Epur. Eaux d'Egout (Russ.), 1926, [8], 9—83).—The results are given of laboratory investigations during the period 1918—1921 on the methane fermentation of sewage sludge at the Moscow sewage disposal works. In purifying plant of the Emscher type the normal alkaline fermentation yields a sludge with properties dependent on the course followed by the biochemical decomposition. The chemical character of the process is influenced by the composition of the fermenting material and its relation to the mass of the sludge; the biological and physiological peculiarities of the micro-flora present, which produce the liquefaction of insoluble organic matter and the decomposition of fatty acids into methane and carbon dioxide; and the pronounced buffering of the liquid, the value of p_H being about 7·8. The general results are in agreement with those obtained empirically, such as the importance of thorough mixing, the influence of temperature, etc. T. H. POPE.

Determination of humus in sewage sludge. BACH (Gesundheitsing., 1926, **49**, 19; Chem. Zentr., 1926, II., 630—631).—The manurial value of the sludge does not depend only on nitrogen, potash, phosphoric acid, etc., but also on the organic matter capable of forming humus, on the physical character, and on the abundance of bacteria. The author designates as "sludge-humus" that constituent of the sludge which effects an improvement in the physical character of soils, and in order to estimate this it is necessary: (*a*) to remove inorganic salts, (*b*) to remove oils and fats, and (*c*) to determine the amount of cellulose, coal, and other substances useless for fertiliser purposes and to subtract this from the remaining dry material. 100 g. of the moist sludge are intimately mixed with 1 litre of distilled water, allowed to settle, and the water is decanted; these operations are repeated twice more. The sludge after drying on the water bath is then extracted with ether and the residue weighed. Of this residue, finely powdered, 5 g. are boiled in a porcelain basin for 30 min. with 250 c.c. of 1·25% sulphuric acid. The liquid, after settling, is poured off into a sedimentation vessel, the residue being twice boiled with 100 c.c. of distilled water, and the water decanted. The residue in the basin is boiled for 30 min. with 250 c.c. of 1·25% potassium hydroxide, the liquid being decanted as before. On the next day the sediment in the two vessels is collected, the residue in the basin is added, and the whole is washed thoroughly with hot water, dried, and weighed. This material can be regarded as useless for the soil, the humus organic material having been dissolved out by the acid and alkaline treatment. The content of humus in sewage sludge from the Ems district was found to be 27·4 to 49·0% in fresh sludge, 27·7 to 43·4% in decomposed sludge. W. G. CAREY.

Colorimetric determination of free chlorine in air. PORTER.—See A., Sept., 927.

Germicidal value of Australian essential oils and their constituents. PENFOLD and GRANT.—See XX.

PATENT.

Utilisation of the effluent from fuller's earth and similar factories. E. MAAG (G.P. 428,486, 3.3.25).—To the mixture of decomposed clay or earth with the effluent, or to the separated effluent, substances such as wood refuse, peat, sulphite-cellulose, etc., are added, and the mixture is boiled, under pressure if desired. Acetic acid and methyl alcohol are produced and the organic substance undergoes degradation to charcoal, which can be used as an absorbent or as a fuel. W. G. CAREY.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCTOBER 15, 1926.

I.—GENERAL; PLANT; MACHINERY.

Oil-burning equipment for industrial furnaces. M. H. Mawhinney (Trans. Amer. Soc. Steel Treat., 1926, **9**, 99—110).—The installation and operation of oil-burning equipment is reviewed. Heating coils should be at the bottom of the tank and the tank connexions of adequate dimensions. The pump must be provided with an air chamber to eliminate pulsations and give a steady oil pressure. The oil viscosity for best atomisation is about 8° Engler. For steady air pressure a turbo-blower is recommended. The burner holes should be flared towards the inside of the wall. The size and location of flues must be such that the products of combustion do not collect in pockets. Baffle-walls should be placed in front of the burners to assist complete combustion. Unsatisfactory heating is generally connected with the size and location of the flues, the size of the combustion space, excessive radiation, or the arrangement of the baffles.

T. H. Burnham.

Measurement of surface temperatures. II. Comparison of various methods. F. W. Adams and R. H. Kean (Ind. Eng. Chem., 1926, **18**, 856—857).—A thermo-couple compensated for heat losses (cf. Boyer and Buss, B., 1926, 647) is the most accurate method for determining the temperature of a hot surface (100—150°), being correct to within 1°, and may be applied to the measurement of the temperature of metallic or non-metallic surfaces, either stationary or moving, such as boiler settings, lagging, moving sheets of pulp, etc. For calculating the heat loss from a surface, results accurate to within 10% are obtained if the temperature is determined by a thermo-couple embedded in the surface, a couple soldered to the surface, a couple consisting of wires connecting through the surface, or a couple held under a pad against the surface. B. W. Clarke.

Factors influencing sedimentation. C. S. Robinson (Ind. Eng. Chem., 1926, **18**, 869—870).—A modified form of Stokes' law, viz., $dH/d\theta = ka^2 (D - d)/z$, where H is the height of the sludge, θ the time of settling, k a constant, a the average dimension of the particles, D the specific gravity of the particles, d the specific gravity of the suspension, and z the relative viscosity of the suspension, is applicable to the settling of fine sludges, as shown by observations with sludges of fine silica particles. B. W. Clarke.

Catalysis—an industrial development. H. S. Taylor (Ind. Eng. Chem., 1926, **18**, 958—960).

Fifty years of development of compressed gases. G. O. Carter (Ind. Eng. Chem., 1926, **18**, 954—956).

Patents.

Filters. Seitz-Werke G.m.b.H. (E.P. 249,857, 19.3.26. Conv., 25.3.25).—The apparatus comprises a tank containing filter-leaves, which are capable of being removed individually through a side door in the tank. The filtrate is drawn off from the leaves at the same side as the door, from both top and bottom.

B. M. Venables.

Atomising and mixing liquids and gases. E. Luscher (E.P. 254,130, 7.8.25).*—Referring to the figures, B, B^1, B^2 are outlets from the rotor, A, which

FIG. I.

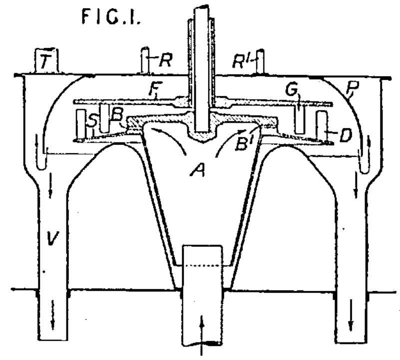
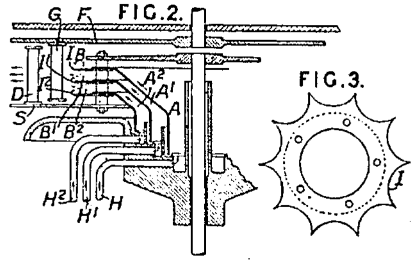


FIG. 2. FIG. 3.



revolves at a high speed. Deflecting plates, G, are attached to a rotor, F, rotating at a different speed, usually in the opposite direction to A. Outer deflecting plates, D, revolve with the rotor, A. In Fig. 2 the rings forming the walls of outlets B, B^1, B^2 are preferably star-shaped, as shown in Fig. 3, the points of the stars being bent alternately upwards and downwards as indicated by arrows, I, I^1, I^2. B. M. Venables.

Furnaces [for pulverised or liquid fuels]. L. C. Harvey (E.P. 255,610, 22.6.25).—A preliminary combustion chamber for a U-shaped furnace burning pulverised solid, or liquid, fuel is provided with one or more longitudinal rows of vertical burners projecting a mixture of fuel and insufficient air downwards. One or more horizontal burners are also provided in the front of the furnace. These burners are lit first and are supplied with enough air for complete combustion of the fuel fed to them. The heat thus generated serves to gasify

the fuel from the vertical burners and combustion is completed by air admitted through the walls of the preliminary combustion chamber, also if desired through the ash-pit which is situated at the bottom where the gases turn upwards to the other limb of the U.

B. M. VENABLES.

Apparatus for heating, cooling, or drying materials. V. CANO (U.S.P. 1,592,078, 13.7.26. Appl., 10.9.25).—An elongated casing is divided by a horizontal partition. The articles to be treated are conveyed through the upper compartment, and a source of heat (or cold) is situated in the lower compartment. A circulation of air is maintained by a blower drawing air through the lower compartment and returning it through the upper compartment in the same direction as the goods travel. B. M. VENABLES.

Drying apparatus. F. G. HATCH (E.P. 255,724, 6.1.26).—Air or other medium which has been used in a drying apparatus is passed through a U-shaped passage in which it passes across five groups of tubes in succession, viz., the first half of a heat interchanger (for cooling), the first half of a refrigerator, around the bend of the U, the second half of the refrigerator, the second half of the heat interchanger (for warming), and a heater. In the heat interchanger and the refrigerator, respectively, the same fluid passes through both sets of tubes. Condensed moisture is collected from the refrigerator compartments and the air is delivered from the heater hot and dry for re-use. B. M. VENABLES.

Drying moisture-containing materials. K. REDMAN, Assr. to B. F. STURTEVANT Co. (U.S.P. 1,593,598, 27.7.26. Appl., 24.2.23).—The material is subjected successively to a current of air or gas of high temperature and humidity and to a current of cooler gas. It is allowed to remain in the cooler gas, and is then subjected to a current of air or gas of a higher temperature and a lower humidity than the first current. H. HOLMES.

Drying apparatus. M. M. MINTER (U.S.P. 1,594,316, 27.7.26. Appl., 23.7.25).—Drying tunnels with end doors are each provided with an air inlet at one end and an outlet near the opposite end. A supply flue communicates with the air inlets by individually-controlled dampers. Air is discharged into the flue from the lower portion of a chamber having a fan mounted in its upper portion. An upper intake opening in the side of the fan chamber communicates with the upper portion of a riser and the lower portion of the riser communicates with an intake flue. A normally-closed opening is provided in the upper portion of the riser for communication with the atmosphere. H. HOLMES.

Apparatus for finely dividing and drying fluid substances. Reducing heavy liquid substances to dry finely-powdered form. (A) J. M. MACLACHLAN, (B) J. C. MACLACHLAN (U.S.P. 1,594,064—5, 27.7.26. Appl., [A] 19.1.22, [B] 2.8.20).—(A) The material to be dried is sprayed centrifugally in the upper part of a chamber. A stream or streams of fluid for disintegrating purposes enters above the spray and passes vertically downwards through it. A drying medium enters the chamber through side inlets. The bulk of the dry powdered material is removed through an outlet at the bottom, and the drying medium with some suspended powder is exhausted through a higher outlet and passes to a separator (*e.g.*, a cyclone) where further dry material is collected. In (B) constructional details of a similar device are given, steam and air being used as the disintegrating and drying medium respectively.

B. M. VENABLES.

Decolorising, purifying, and adsorbent composition. J. K. STEWART, Assr. to SHELL Co. OF CALIFORNIA (U.S.P. 1,592,543, 13.7.26. Appl., 27.2.24).—Sodium silicate and a solution of an aluminium salt are mixed and the precipitate is dried, ground, and washed to remove the sodium salts. The resulting product contains only silica and alumina and is used as a decolorising agent when ground to 200-mesh and as an adsorbent when ground to 40-mesh. The product is activated by heating to 200°.

E. S. KREIS.

Extraction apparatus. R. P. SOULE, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,594,024, 27.7.26. Appl., 11.6.24).—A vessel closed for operation at pressures above atmospheric is divided into a mixing chamber and a settling chamber. Means are provided for introducing the raw material and the solvent into the mixing chamber, for agitating them while maintaining them at an elevated temperature, and for the separate removal from the settling chamber of the solution and the insoluble residue.

H. HOLMES.

Balanced ball mill. W. M. BARKER (U.S.P. 1,594,990, 3.8.26. Appl., 26.5.26).—The mill comprises a feed chamber, a discharge chamber, and an annular ball-race chamber. The material passes through inlet ports from the feed chamber to the ball-race chamber, is ground by the balls therein, and passes through outlet ports into the discharge chamber. H. HOLMES.

Cooling hot salt solutions in a vacuum. KALI-FORSCHUNGS-ANSTALT G.M.B.H., Assees. of E. RITTER (G.P. 430,482, 18.3.25. Addn. to 413,710; B., 1925, 743).—The addition of water to replace that evaporated is made at such places that incrustations during the crystallisation are avoided. B. FULLMAN.

Filter-presses. F. J. BISBEE (E.P. 256,898, 19.4.26). —See U.S.P. 1,589,834; B., 1926, 696.

Apparatus for washing and classifying minerals. C. CLOUWEZ (E.P. 245,770, 6.1.26. Conv., 9.1.25).

Cooling towers. J. M. SEYMOUR (E.P. 256,889, 29.3.26).

Removal of dust, fumes, and the like. J. FORGAN-POTTS and V. R. CHADWICK (E.P. 257,032, 23.5.25).

Heat interchanging apparatus. L. CHAVANNE (E.P. 231,186, 249,534, and 249,536, 19.3.25. Conv., 19.3.24).

Steam distillation (U.S.P. 1,594,957).—See II.

Exothermic gaseous catalytic reactions (E.P. 255,963—4).—See VII.

Separating gas mixtures (U.S.P. 1,594,336).—See VII.

Lining pipes etc. (E.P. 255,546).—See XIII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Lignin and oxycellulose theories [of coal formation]. J. MARCUSSON (Z. angew. Chem., 1926, **39**, 898—900; cf. B., 1925, 386).—The lignin content of coniferous wood remains approximately constant (about 20%) during its decay. The alkali-soluble compounds can be resolved into humic acids, insoluble in water, and a water-soluble portion showing the reactions of glycuronic acid and also apparently containing pentoses. The content of these compounds, both soluble and insoluble in water, increases as decay proceeds, the insoluble acids being partly derived from cellulose, since they occur in combination with carbohydrates. The content of volatile acids, in particular acetic acid, and of methyl alcohol increases with advancing decay. Both lignin and cellulose go to form coal. Cellulose is converted, by hydrolysis and oxidation, into oxycellulose, and pectins are also formed. The glycuronic acid residue in oxycellulose is readily converted into humic acids which, in turn, lead to coal.

W. T. K. BRAUNHOLTZ.

Oil obtained by "berginisation" of Lower Silesian coal. M. HEYN and M. DUNKEL (Brennstoff-Chem., 1926, **7**, 20—25, 81—87, 245—250).—A detailed analysis of the oils obtained from unwashed coal dust containing 17·7% of ash, 28·5% of volatile matter (both reckoned on the dry coal), and an exceptionally high proportion of fusain, which is known to yield very little oil on "berginisation." The coal was made into a paste with half its weight of an oil obtained from a previous "berginisation" of similar coal, and the experiment was run continuously for 2½ days at a throughput of 7 kg. of paste per hr. The total oil yield was 36·45% of the raw coal, *i.e.*, 33·09% of oily product and 3·36% of benzine recoverable from the gas. The black, viscous oil contained 29·5% of dust, to remove which the light oils were distilled off up to 230° (during which ammonium carbonate separated out in the receiver), and the residue was dissolved in benzene and filtered warm. After removing the benzene, the light and heavy oils were united and fractionally distilled (under diminished pressure above 220°) up to pitch (9·26% of the raw coal). It is difficult to remove the oil completely from the dust, which, after extraction, contained 40·65% of ash and 10·45% of volatile matter. The oil fractions were extracted by the usual methods for phenols, bases, etc., showing the total distillate to be composed of 75·7% of neutral oils, 12·0 % of phenols, 3·5% of bases, and small traces of carboxylic acids, resin, and a sediment. 10·3% of the phenolic compounds was phenol itself, whilst phenol, cresols, and xylenols (b.p. up to 220°) constituted 66·5% of the whole. The basic fraction contained primary (aniline, toluidine, xylidine), secondary (not identified), and tertiary bases (pyridine absent; collidine, quinoline, quinaldine, and probably *iso*quinoline identified). The "benzine" from the gas was refined with sulphuric acid and fractionally distilled, the bulk distilling below 185° and containing benzene, toluene, xylene (shown by nitration), with a large proportion of their hexahydro-derivatives (shown by dehydrogenation). The light oil fraction (b.p. up to 180°) of the main product, similarly refined and fractionally distilled, had an analogous composition, the ratio of aromatics to hydroaromatics being, however, greater than in the "benzine." The higher fraction (180—220°) of the oil contained naphthalene and its tetrahydro- and decahydro-derivatives. All the neutral oil fractions were remarkable for their low sulphur content (0·76—1·12%). Attempts to prepare a satisfactory lubricating oil from the higher fractions were unsuccessful. The solid constituents of the neutral oils comprised a small quantity of paraffins and a considerable quantity of aromatic hydrocarbons (mainly phenanthrene). W. T. K. BRAUNHOLTZ.

Combustion of carbonic oxide. W. A. BONE (Gas J., 1926, **174**, 725—731; Gas World, 1926, **85**, 4—6, 34—38).—A brief historical survey of the subject is given, followed by a description of recent investigations carried out under the author's direction (cf. A., 1925, ii., 928; 1926, 8, 480).

Nomenclature of wood distillation products. G. BUGGE (Z. angew. Chem., 1926, **39**, 1032—1033).—In agreement with Mallison ("Teer, Pech, Bitumen, u. Asphalt," Halle, 1926), it is proposed to employ for tars and pitches obtained by distillation of natural organic materials names derived both from the raw material and the method of distillation. The name "wood oil" should be restricted to tung oil. The name creosote should only be used with a prefix denoting the origin.

S. I. LEVY.

Ammonia recovery [from coal gas] by gypsum. H. W. JACKMAN (Gas J., 1926, **175**, 428—429).—In a method worked out on the laboratory scale sulphuric acid for the recovery of ammonia at gas works is replaced by gypsum. Ordinary 15% ammonia concentrate is prepared and is saturated with carbon dioxide obtained from flue-gases in a packed tower. To this solution gypsum is added in small quantities at a time with stirring, the calcium carbonate filtered off, and the liquor passed through a still to remove the remaining free ammonia. The liquor is then evaporated until ammonium sulphate crystallises. A direct process is also possible by washing the gas with saturated calcium sulphate solution in a packed tower. The calcium carbonate sludge is drawn off from time to time and the liquor finally evaporated to crystallisation. The removal of ammonia from the gas is incomplete and depends chiefly on the temperature and content of volatile ammonia of the scrubbing liquor. The residual ammonia is removed by a scrubber fed with water.

C. IRWIN.

Vacuum or steam distillation of benzol [wash-oil]. O. HILGENSTOCK (Brenustoff-Chem., 1926, **7**, 87—89).—As regards the rate of driving off the benzol, and the steam, wash-oil, and cooling water consumption, distillation of wash-oil with steam is preferable to vacuum distillation, and it is not inferior from the point of view of the efficiency of scrubbing of the gas, the composition of the crude benzol, or the corrosion of the iron apparatus. The cause of the thickening of the wash-oil is not yet established. W. T. K. BRAUNHOLTZ.

Oil-bearing shales in North Carolina. F. C. VILBRANDT (Ind. Eng. Chem., 1926, **18**, 793—795).—An examination of the shale in the Deep River Valley coal field leads to an estimate of the presence of one billion tons of oil-bearing shale capable of yielding approximately 27 billion gallons of shale oil (30% of which would be a straight-run motor spirit) and 47,500 tons of ammonium sulphate. The shales are overlying and underlying coal measures which are being worked.
W. N. HOYTE.

Anti-knock motor fuels by cracking shale oil. J. C. MORRELL and G. EGLOFF (Ind. Eng. Chem., 1926, **18**, 801—802).—Shale oil from America, Australia, and France when cracked at 120—150 lb. pressure, yielded more than 50% of Navy end point gasoline; this, by chemical analysis and by actual engine tests, was found to possess anti-knock properties. A suitable scheme of refining with sodium plumbite and sulphuric acid is described. W. N. HOYTE.

Fractionation number [of motor fuels]. W. OSTWALD (Petroleum, 1926, **22**, 850—853).—The characteristic number of the b.p. curve of a motor spirit is obtained by drawing a straight line at right angles to the temperature axis such that the two areas enclosed by this line, the b.p. curve, and the uprights at either end of the curve are equal. The temperature indicated at the intersection of this line and the temperature axis is the characteristic number. In order to express concisely the sharpness of fractionation by which a spirit was prepared a straight line is drawn through the b.p. curve, inclined to the temperature axis and cutting the curve in at least two points, such that the two areas (above and below the line respectively) enclosed by the line, the curve, and the uprights) are equal. The inclination of this line to the horizontal is the fractionation number of the spirit, and when this is used in conjunction with the characteristic number calculations of the composition of mixtures are possible and the efficiency of a fractionating column can be stated. W. N. HOYTE.

Variation of specific gravity of Japanese crude oils. T. IKI (J. Fac. Sci. Tokyo, II, 1926, **1**, 53—64).—Tables are given showing the variations in d of crude oils from different geological beds (the range here being 11° to 48° B; d 0·993—0·786), from the same bed, from different depths, when under the influence of volcanic activity, and after exposure to the air. The causes of these variations are discussed.
L. J. SPENCER.

Composition of Mid-continent petroleum. C. F. MABERY (Ind. Eng. Chem., 1926, **18**, 814—819; cf. B., 1924, 120).—Details of fractional extraction of crude and lubricating oils with ether and alcohol are given. Two series of compounds were separated, viz., the "D hydrocarbons" (lubricants), and the "H hydrocarbons" (asphaltic oils). Molecular weight determinations showed that the series C_nH_{2n-4} and C_nH_{2n-8} are the best lubricants; those poorer in hydrogen show asphaltic properties. Marked differences were observed between the refractive indices of the D and H series, the former having the higher index. Viscosities of the fractions were determined in Ostwald tubes and the resistance of films of the oils to stress in a hard Babbitt-metal bearing. W. N. HOYTE.

Specific heats of oils. A. R. FORTSCH and W. G. WHITMAN (Ind. Eng. Chem., 1926, **18**, 795—800).—The apparatus used consisted of a large vacuum flask, the contents of which were rapidly stirred and heated by an immersed resistance coil. The outside wall of the flask was surrounded by an air bath maintained at the average temperature at which the determination was carried out. Radiation corrections were applied. Observations were taken over temperature intervals from 50° to 430° F. (10° to 220°). Fifteen oils from Mid-continent crude oil were studied; their specific gravities ranged from 0·75 to 1·00. It was found that specific heats for engineering purposes could be calculated from the formula $C = (t + 670)\ (2 \cdot 10 - d)/2030$, where C = specific heat, t = temperature in ° F., and d = sp. gr. at 60° F. The maximum deviation for any oil studied was 5·9%; the average deviation was 2·3% at 240° F. The equation cannot be applied without further correction to oils within 100° F. of their critical temperature. A review of the results of other workers is given. W. N. HOYTE.

Cracking of petroleum oils. E. H. LESLIE and E. H. POTTHOFF (Ind. Eng. Chem., 1926, **18**, 776—785).—The cracking of various petroleum oils has been studied under controlled conditions on the laboratory scale. The apparatus consisted of a preheater in which the oil, about 3 litres, is rapidly brought to the desired temperature by circulation over electrically heated coils, and from which it is discharged into a bomb surrounded by resistance coils whereby heat losses from the bomb can be made good. Special precautions are taken to avoid skin temperature overheating. Three oils were studied, a gas oil, a heavy fuel oil, and a "thermolised" gas oil, *i.e.*, one already cracked and the spirit removed. The rate of cracking was found to double for a rise in temperature of 12°. At 370° the thermolised gas oil polymerised at a greater rate than it cracked.

The formation of oils of low boiling point was found to be a straight line function of time with the above exception of the thermolised gas oil, and can be formulated as a reaction of the first order. Pressure as such has no effect on the percentage or boiling range of the spirit obtained. Increase of pressure decreased the percentage of unsaturated compounds in the spirit. The fuel oil cracked most easily, the thermolised gas oil the least easily. Removal of the spirit as formed had no effect on the percentage obtained; the spirit, if removed progressively, is, however, somewhat more unsaturated. Under no circumstances was a condition of equilibrium attained between the products of the decomposition of the oil. The spirit from the thermolised gas oil for a given boiling range had the highest specific gravity. Experiments at atmospheric pressure in a special apparatus showed that cracking was an endothermic reaction to the extent of approximately 500 cal. per g. of spirit produced. This emphasises the importance of efficient heat insulation of reaction vessels in full-sized cracking plants.
W. N. HOYTE.

Development of a liquid-phase cracking process. S. J. M. Auld and A. E. Dunstan (Ind. Eng. Chem., 1926, **18**, 803—807).—The theoretical considerations and practical experiments on which the A.D.H. cracking process is based are outlined. The pressure–solubility relations of the permanent hydrocarbon gases and cracking stock oils were examined in a modified Andrews apparatus in which the oil could be heated to 450° under pressures up to 1000 lb. per sq. in. and could be kept under observation in a silica tube. Examination of the time factor in relation to the extent of cracking was carried out in an autoclave. It was found that the higher the pressure the less unsaturated is the resulting cracked spirit and the less is the formation of carbon. High pressures induced by the adsorption of gas oil in activated charcoal maintained at a suitable temperature produced 70% of superior motor spirit. The formation of carbon in the cracking process is primarily due to the thermal decomposition of olefinic compounds. The reduction to a minimum of the superheating of the oil on the surfaces of the plant is emphasised. Persian gas oil was found to begin to crack at 380°, and the rate of cracking to double, other things being equal, for each 10° rise in temperature. As the result of experiments carried out in electrically heated steel tubes it was decided that the oil should be heated while in narrow tubes and moving with low velocity in order to prevent excessive skin temperature in the heating walls. Three definite zones were established in the plant designed, viz., the preheaters, where the oil is brought to a temperature some 20° below cracking temperature; the thermolyser, where cracking temperature is slightly exceeded; and the reactor, where the oil remains sufficiently long at cracking temperature for the desired amount of cracking to take place. Previous to the formation of coke in the cracking plant the oil passed through a pitch-like, viscous state, and it was possible to draw off progressively this substance as formed, thereby preventing the formation of coke to a large extent. W. N. Hoyte.

Determination of unsaturated content of petroleum products. A. W. Francis (Ind. Eng. Chem., 1926, **18**, 821—822).—A method is described in which the oil is shaken with an aqueous mixture of potassium bromide and bromate in slightly acid solution. Bromine is generated slowly and is fixed at the double linkings in the unsaturated compounds present, substitution being reduced to a minimum. Results on synthetic mixtures of pure unsaturated compounds in straight-run motor spirits show very good agreement between observed and calculated figures. W. N. Hoyte.

Desulphurising effects of sodium hypochlorite on naphtha solutions of organic sulphur compounds. A. E. Wood, A. R. Greene, and R. W. Provine (Ind. Eng. Chem., 1926, **18**, 823—826).—The desulphurising effect of sodium hypochlorite on naphtha solutions of ethyl sulphide, *n*-butyl sulphide, phenyl sulphide, ethyl disulphide, *n*-propyl sulphide, ethyl mercaptan, *n*-propyl mercaptan, *iso*amyl mercaptan, carbon disulphide, hydrogen sulphide, thiophen, and elemental sulphur was determined. The effect was found to depend on the actual sulphur compound used, the amount of available chlorine present, the degree of alkalinity of the hypochlorite solution, and the time and intensity of agitation. Thiophen and elemental sulphur are not attacked by sodium hypochlorite; the aliphatic sulphides vary in the ease with which they are oxidised to sulphones; the disulphides are somewhat less readily attacked than the sulphides, and the mercaptans still less readily. Hydrogen sulphide is oxidised to elemental sulphur, and carbon disulphide is partially removed. The conversion of the sulphides into sulphones is beneficial in refinery practice, as the latter compounds are more easily removed from the naphtha by subsequent treatment, re-distillation, and filtration through an adsorbent material. Better results in general are obtained by using hypochlorite solutions comparatively rich in chlorine and alkali rather than solutions weak in these respects. In designing a suitable hypochlorite treatment for a particular naphtha, a careful study must be made as regards the oxidation products; if these are acidic, then sufficient alkali must be present initially to maintain a final alkaline solution. W. N. Hoyte.

Distillation of lubricating oils under high vacuum. B. T. Brooks (Ind. Eng. Chem., 1926, **18**, 789—793).—A brief review of the previous work on the large scale is given. Details of the work of Schulze on the refinery scale in the United States are given. A vacuum of 5—3 mm. is used, and a wax-free oil is distilled. The distillate is emulsified with just sufficient strong caustic soda solution to combine with the naphthenic acids, and a second high-vacuum distillation then yields a lubricating oil requiring no further treatment. Cuts of very narrow boiling range can be made, and oils with viscosities up to 175 sec. Saybolt at 99° can be obtained. W. N. Hoyte.

[Paraffin] wax crystallisation. F. W. Padgett, D. G. Hefley, and A. Henriksen (Ind. Eng. Chem., 1926, **18**, 832—835).—The crystallisation of various waxes from solution in oil was studied by microscopical examination of samples under crossed Nicols. The effect of the viscosity of the solvent, the rate of cooling, and time elapsing after crystallisation was complete, was studied. Three types of crystallisation were detected, viz., very small crystals from residual oils and petroleum, fine needles from slack wax and ordinary paraffin wax, and plate-like crystals produced in small numbers during the initial crystallisation of paraffin wax, and also deposited between the above-mentioned needles on keeping for some time. W. N. Hoyte.

Oxidation of coal at storage temperatures, S. W. Parr and R. T. Milner (Fuel, 1926, **5**, 298—300). —See B., 1925, 195.

Deterioration and spontaneous combustion of coal in storage. S. W. Parr (Fuel, 1926, **5**, 301—305). —See B., 1925, 195.

Carbon dioxide as an index of critical oxidation temperature for coal in storage. S. W. Parr and C. C. Coons (Fuel, 1926, **5**, 306—308).—See B., 1925, 195.

Use of oxygen in manufacture of water-gas. F. E. Vandaveer and S. W. Parr (Fuel, 1926, **5**, 309—314).—See B., 1925, 979.

Fifty years of gas chemistry. W. H. Fulweiler (Ind. Eng. Chem., 1926, **18**, 945—948).

Fifty years of wood distillation. L. F. Hawley (Ind. Eng. Chem., 1926, **18**, 929—930).

Fifty years in the petroleum industry. F. A. Howard (Ind. Eng. Chem., 1926, **18**, 936—938).

Oil-burning equipment for industrial furnaces. Mawhinney.—See I.

Fluorescence of oils in ultra-violet light. Croner. —See XII.

Patents.

Coal washing. W. C. Menzies (U.S.P. 1,594,626, 3.8.26. Appl., 23.9.24).—Coal is fed on to an inclined grid which forms the false bottom of a tank. A stream of water is directed on to the coal from the under-side of the grid and takes the lighter clean coal into suspension. Two baffles supported at an adjustable distance above the grid, and extending over the whole width of the tank, direct the flow of the suspension. The upper edge of one of the baffles forms a weir over which the coal and water flow into a separate compartment, where the clean coal is separated. The heavier coal constituents are not lifted sufficiently by the water-stream to carry them over the weir, and they gravitate down the inclined grid, being separately collected at the bottom. S. Pexton.

Agglomerated combustible products. E. Goutal and H. Hennebutte (E.P. 247,210, 5.2.26. Conv., 9.2.25).—The agglomerated products resulting from baking blends of low-temperature coke and pyroligneous tars and pitches or their products at a medium temperature (cf. E.P. 231,863, B., 1925, 906) disintegrate when heated to a high temperature. This defect is obviated by incorporating in the blend suitable proportions of hard coals or other combustible materials which do not shrink when heated to high temperatures. The compressed moulded blends are baked at 600—700°, and the product, being dense and highly combustible, is suitable for burning in an open grate or in a suction gas producer, where retention of the original shape of the fuel is advantageous for uniform working. S. Pexton.

Briquette. H. McC. Spencer, Assr. to Seydel Chemical Co. (U.S.P. 1,590,706, 29.6.26. Appl., 17.1.23). —Dry anthracite culm, coal dust, or the like mixed with 1—10% of phosphoric acid in 50% solution in water is briquetted and dried at 220°. A hard briquette which resists the action of water is obtained. In place of phosphoric acid, a soluble phosphate, *e.g.*, the extract obtained from phosphate rock by treatment with sulphuric acid, may be used. The method may also be applied to briquetting iron sulphide ores. T. S. Wheeler.

Carbonising coal. Internat. Combustion Engineering Corp., Asses. of W. Runge (E.P. 253,878, 19.5.26. Conv., 17.6.25. Addn. to 242,621).—Pulverised coal is allowed to gravitate through a retort up which air preheated to approximately 425° is passing at such a velocity that the coal is buoyantly held within the retort for 60 sec. The treated coal deprived of its agglutinating power retains its original form and most of its volatile matter. In this form it is mixed with a proportion of untreated coal and carbonised, giving a finely-powdered coke, the content of volatile matter in which can be regulated by varying the carbonising conditions. S. Pexton.

Producing coke. M. J. Trumble (E.P. 255,620, 2.7.25).—Carbonaceous material is carbonised in an externally heated retort, at a medium temperature, and superheated steam is passed through the charge until it is free from volatile matter. The retort temperature is then further raised and oil is introduced into the incandescent coke. The oil is cracked to light petroleum and a pitchy residue, which is deposited in the voids of the coke and converts it into a consolidated fuel of high calorific value. By regulating the conditions of cracking the volatile content of the coke can be adjusted to satisfy the special purpose for which the fuel is required. S. Pexton.

Carbonising coal. R. L. Rodgers (E.P. 256,038, 15.7.25).—Coal is carbonised at a low temperature in narrow metal chambers, which have double walls. The space between the walls is filled with a fusible salt, *e.g.*, a mixture of 75% of calcium chloride and 25% of sodium chloride. The chambers each have tight-fitting charging and discharging doors, and several are mounted in a direct-fired furnace. The flames do not cause local overheating of the coal charge because of the interposition of the salt bath, which also by reason of its large thermal capacity materially increases the rate of carbonisation in the early stages. S. Pexton.

Apparatus for carbonising coal. Carbonising process. C. Hayes (U.S.P. 1,593,448—9, 20.7.26. Appl., 22.5.26. Conv., 16.5.25).—Crushed coal or lignite is treated in a preheater at 205° to expel moisture, and then conveyed to the top of two digesters enclosed by a brick-lined chamber, in which the gases generated are collected. A plunger fits into the top of each digester, the lower part of which is heated to 430°, the upper part to 315°, by means of oil burners arranged outside. Superheated steam is also passed through the charge. When the digester has been completely filled, the plunger is forced down to expel air. When the coal in the lower portion of the digester becomes converted into a mass of softened coke-like material from which most of the volatile products have been removed, but leaving just sufficient to make it ignitible, the bottom of the digester is opened and the softened coke is forced out into a bin underneath where the heat lost during cooling is utilised to generate steam for the plant. The charge in the upper part of the digester descends, and its place is taken by fresh coal from the preheater. The operations are then repeated. R. B. Clarke.

Manufacture of calcium carbide, coke, and synthetic products [methyl alcohol]. G. L. E. Patart (F.P. 605,530, 9.10.25).—Coke is prepared by the carbonisation of coal in an oven or retort which communicates directly with a calcium carbide furnace, thus facilitating the introduction of coke into the latter a

the highest temperature reached during its formation. The distillation products from the coal are mixed with carbon monoxide and catalysed under pressure for the production of synthetic methyl alcohol. R. B. CLARKE.

Synthetic bone char from black-strap syrup. C. B. DAVIS (U.S.P. 1,592,599, 13.7.26. Appl., 12.5.23).—125 lb. of commercial superphosphate are extracted with water, and the solution is mixed with 1 ton of black-strap syrup and hydrated lime added until alkaline. The gelatinous mass which precipitates carries with it the albuminous and gummy matter present in the black-strap. It is filtered off and carbonised at 500° in the absence of air. A synthetic bone char which is very effective in the purification of sugar solutions is obtained. T. S. WHEELER.

Apparatus for making water-gas. R. R. and J. S. McDONNELL (U.S.P. 1,592,464, 13.7.26. Appl., 26.3.21).—The generating chamber has a grate containing passages connected with a pipe through which water is supplied for the purpose of generating and superheating steam. The steam is discharged through an outlet below the grate and passes upwards, through the grate, to the fuel bed. W. T. K. BRAUNHOLTZ.

Gas-producing apparatus. E. L. BROOME, Assr. to GEN. REDUCTION GAS & BY-PRODUCTS CO. (U.S.P. 1,590,279, 29.6.26. Appl., 17.1.21).—An apparatus for gasifying slack, sawdust, or other waste fuel consists of a conical vessel rotating about its axis, which is slightly inclined to the horizontal. The fuel is introduced at the upper and narrow end, and the clinker, which is formed into balls by the rotation, is withdrawn at the lower end by a number of extractors, each of which consists of a tube bent twice at right angles through which the spherical clinker masses pass more readily than the fuel. Air or steam is introduced into the interior of the mass of fuel by a water-cooled tuyère. To improve the admixture of vapour and solid fuel the cylindrical vessel is fitted internally with a baffle perpendicular to its axis, which divides it into a drying and a reaction chamber. T. S. WHEELER.

Manufacture of gas. ANC. ÉTABL. BARBIER, BÉNARD, and TURENNE (SOC. ANON.) (F.P. 605,407, 22.10.25).—Animal, vegetable, or mineral oils are cracked at 600—700° in one chamber of a retort having a second chamber on the same level. In this second chamber the vapours are heated to 900—1000°. They then pass to a second retort at a temperature of 500°, this retort being charged with iron borings. C. IRWIN.

Aeration and gas purification apparatus and methods. Aeration and gas purification processes and apparatus. Gas purification processes and apparatus. Gas and liquid contact apparatus and methods. Purification of gases. Removal of hydrogen sulphide and other noxious constituents from gases. Purification of gases. W. J. MELLERSH-JACKSON. From KOPPERS CO. (E.P. 255,139—255,145, 14.4.25).—(A) In the process of removing hydrogen sulphide from gas by washing with a suspension of iron oxide in a solution of sodium carbonate, the spent liquor is regenerated by bubbling air through it in a "thionizer." Difficulty has been experienced through the blockage of the pores in the air bubblers with sediment. This has been overcome by surrounding the aeration tubes with an envelope of pliable porous material, such as cotton, which is free to vibrate; no sediment then adheres to the envelope and the aerator remains free. (B) By maintaining the absorber and thionizer in the wet purification process at a temperature above 29·5° instead of 15·5°, the following advantages result:—The capacity of a given plant is doubled; it is no longer necessary to use freshly-precipitated iron hydroxide, as finely-powdered bog-ore gives excellent results; and the sulphur in the thionizer separates as a relatively heavy sludge rather than as a bulky foam. (C) In the aeration of spent washing liquor secondary reactions such as the formation of sodium thiosulphate occur. Such reactions are greatly retarded if the spent liquor entering the thionizer is neutral or slightly acid and of low concentration. To attain this and yet completely purify the gas two absorbers, each connected with separate compartments of a thionizer, are used. The first absorber, which removes the bulk of the hydrogen sulphide, is fed with a suspension of 1% of iron oxide in a 1% solution of sodium carbonate. Renewals of the iron in this circuit may be made by adding acid salts such as the chloride or sulphate to the spent liquor entering the thionizer. When the sulphur in the thionizer connected to the first absorber becomes excessive the first absorber is connected with a second chamber of the thionizer containing regenerated liquor of 1% alkalinity, and the accumulated sulphur is worked up. In the second absorber a 1% suspension of iron oxide in a 3% solution of sodium carbonate is used, but because of the low concentration of hydrogen sulphide in the gas entering the second absorber this liquor can be regenerated without material loss of sodium carbonate. (D) An improved design of contact material for filling gas absorption apparatus is described. It consists of alternate series of distributors and fillers. The distributors comprise a number of surfaces inclined to the direction of liquor and gas flow, whilst the filler surfaces are parallel to the liquor and gas flows. (E) A wet purification process in which the washing medium is a 1% solution of sodium carbonate charged with 3% of finely divided iron oxide, is described. The hydrogen sulphide absorbed by the sodium carbonate reacts with the iron oxide forming iron sulphide in which form the bulk of the sulphur is removed. The iron sulphide is separated from the liquor and oxidised as a sludge in an aerator, free sulphur and iron oxide being formed. This regenerated mixture is filtered and returned with the clear liquor to the washing system. From time to time some of the oxide after regeneration is withdrawn for the recovery of sulphur, and the oxide returned to the system. (F) Foul gas is washed with a 2% suspension of magnesium oxide in a 0·5% solution of magnesium chloride. The spent liquor is heated and agitated, whereby hydrogen sulphide is evolved. The hydrolysis of the hot magnesium chloride with the production of hydrochloric acid assists the liberation of the hydrogen sulphide. The revivified suspension is used for washing more foul gas. (G) When purifying gas with aqueous suspensions

of metallic compounds the regeneration is rendered more efficient if a peptising agent such as glycerin is added to the washing medium in order to prevent coagulation of the solid particles of the reagent.

S. PEXTON.

Separation of hydrocarbons from earthy matter. J. M. MCCLAVE, Assr. to BITUMINOUS SAND CO. (U.S.P. 1,594,625, 3.8.26. Appl., 27.11.22).—The addition of a colloidal clay to a suspension of earthy matter containing hydrocarbons causes the latter to separate and become recoverable. S. PEXTON.

Treatment of hydrocarbon oils [crude benzene etc.]. H. SCHLAMP (E.P. 255,777, 30.3.26).—The crude benzene recovered from the distillation of gases from coal, lignite, peat etc., or a corresponding product in the purification of petroleum, is, if necessary, freed from phenols by washing with alkali and from pyridine etc. by washing with dilute sulphuric acid, and is then treated with concentrated sulphuric acid. A neutralising medium such as ammonia solution or alkali carbonate is added, and the heat of reaction effects the distillation of the lighter hydrocarbons. Carbon dioxide liberated may be used for the separation of phenols from the soda-phenol lye. On keeping, the reaction mixture separates in layers; to recover the remaining volatile hydrocarbons only the uppermost layer has to be distilled. The operation is carried out in one vessel provided with stirring devices, and the products of neutralisation are of commercial value. H. MOORE.

Treating carbon and carbon compounds under heat and pressure. F. BERGIUS (U.S.P. 1,592,772, 13.7.26. Appl., 30.8.21. Conv., 23.1.15).—The reagents are heated to within 50° of their reaction temperature before introduction into the pressure chamber, which is heated by circulating within its double walls an inert gas under the same pressure as the reagents, so that the heat-conducting wall need not be built thick to withstand pressure. E. S. KREIS.

Oil-treating [cracking] apparatus. E. A. DIETERLE (U.S.P. 1,589,631, 22.6.26. Appl., 7.4.23).—Oil is sprayed into the top of a cylindrical shell containing a number of S-shaped flues through which hot gases are passed. The oil first falls on to the cooler portions of the flue and then passes on to the hotter portions, and is thus gradually heated and cracked. Uncracked oil is withdrawn from the bottom of the shell. The apparatus is safe to handle as only a relatively small quantity of oil is under treatment at one time. T. S. WHEELER.

Oil-treating apparatus. J. W. LEWIS, Assr. to ATLANTIC REFINING CO. (U.S.P. 1,593,905, 27.7.26. Appl., 6.7.22.).—A pressure still comprises a vertical still combined with a tubular heat absorption structure. The upper and lower ends of the tubes are connected to the still at different levels, and the connexions may be closed by valves which may be operated simultaneously. Pressure above atmospheric is maintained in the still and tubes. An oil chamber is also connected with the heat-absorption structure by a valved connexion, with by-pass and a check valve in the by-pass. Pressure above atmospheric is maintained in the oil chamber.

H. MOORE.

Removal of sulphur and sulphur compounds from hydrocarbon-oil distillates. G. L. WENDT, Assr. to STANDARD OIL CO. (U.S.P. 1,594,083, 27.7.26. Appl., 16.7.24).—Distillates containing products within the gasoline and kerosene b.p. range are treated with "doctor" solution, and then distilled at atmospheric pressure and at a temperature not above 205°. The distillation is continued under reduced pressure without raising the temperature. H. MOORE.

Cracking hydrocarbon oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,594,093, 27.7.26. Appl., 21.10.22).—The oil is raised to cracking temperature in a heating zone and then collected in a vaporising zone, where vaporisation takes place under pressure. The vapours pass to a dephlegmating zone, where they are subjected to the refluxing action of the entering raw oil. The uncondensed vapours pass under reduced pressure to a series of fractionating zones, the vapours from the last of which are condensed. Portions of the condensate are returned from each fractionating zone to the preceding one. H. MOORE.

Steam distillation [of hydrocarbon and like liquids, immiscible with water]. F. A. HOWARD and E. M. CLARK, Assrs. to STANDARD DEVELOPMENT CO. (U.S.P. 1,594,957, 3.8.26. Appl., 8.3.20).—The liquid is passed through a series of stills in which successively heavier fractions are removed. Steam is introduced into the later stills of the series and the vapours withdrawn therefrom, containing at least 50% of steam, are compressed. The compressed vapours are introduced into the stills in which the lighter fractions are removed and the vapours from these stills are removed and condensed. H. HOLMES.

Conversion of hydrocarbons [into products of lower boiling point]. V. LOUKINSKY and W. ROBIKOFF (F.P. 606,233, 19.2.25).—Gas oil or petroleum is converted into light hydrocarbons by the catalytic action of sodium aluminium chloride, $NaAlCl_4$, or of a compound of aluminium chloride, alumina, and silica.

C. IRWIN.

Dehydrating crude oil. R. J. BARRY (U.S.P. 1,593,893, 27.7.26. Appl., 4.12.25).—Emulsions of oil and water are separated by adding small quantities of salt and diatomaceous clay and heating the mixture.

H. MOORE.

Purifying [used lubricating] oil. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of C. G. VAN BRUNT (E.P. 242,272, 26.10.25. Conv., 29.10.24).—Oil containing suspended matter, *e.g.*, used lubricating oil, is treated with feebly basic hydrophile material, such as sodium silicate, in a vessel provided with mixing devices. Thence the oil passes to a settling tank containing heated water. From this the oil passes by a weir overflow to an evaporator where diluents are removed. The silicate solution, in a state of fine division, forms a sludge with the impurities, and this sludge subsides

into the hot water. The operation is conducted at a temperature of 70—80°, and the purified oil gives up its heat to the incoming oil in a heat exchanger.

H. MOORE.

Making coal briquettes of anthracitic quality from immature carboniferous substances. J. BEAUDEQUIN (U.S.P. 1,597,570, 24.8.26. Appl., 6.6.23. Conv., 7.6.22).—See E.P. 216,691; B., 1924, 702.

Manufacturing [fuel] briquettes. J. BEAUDEQUIN (U.S.P. 1,597,571, 24.8.26. Appl., 29.11.24. Conv., 3.12.23).—See E.P. 225,849; B., 1926, 349.

Powdered fuel or gas burner. A. L. MOND. From H. T. DYER (E.P. 256,686, 12.5.25).

Dry-quenching of coke. WOODALL-DUCKHAM (1920), LTD., and J. W. REBER (E.P. 256,727, 25.5.25).

Lining pipes etc. (E.P. 255,546).—See XIII.

III.—TAR AND TAR PRODUCTS.

Determination of anthracene. K. KOCH (Z. anal. Chem., 1926, 68, 359—362).—See Jacobsohn, B., 1926, 734.

PATENTS.

Distillation [dehydration] of tar and the like. W. B. DAVIDSON, A. C. MICHIE, and E. W. MUDDIMAN (E.P. 255,919, 1.4.25).—Frothing, which is a serious difficulty, especially in the distillation of low-temperature tars, may be prevented by the provision of a heated surface above the level of the liquid tar. A tar dehydrator which is heated by means of a horizontal steam coil just below the surface of the tar can be modified by the provision of a further series of closely pitched steam tubes in the vapour space. These are fed with steam at 120—160°, which then passes on to the lower coil. Should frothing occur, the froth is broken, and in addition the cooling of the steam results in the rate of heating of the still being automatically slowed. Crude tar, without preheating, enters the still at a low level on one side, and dehydrated tar is drawn off from the upper part on the other side, the process being continuous.

C. IRWIN.

Treating wood tar oil. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,590,156, 22.6.26. Appl., 27.3.24).—Tar oil from the distillation of wood is saturated with ammonia and is heated in an autoclave under a pressure of 7 atm. On distillation a good yield of a light-coloured oil, with a pleasant odour, boiling up to 125°, is obtained. It is of value as a solvent for nitrocellulose. A similar product is formed by treating the tar oil with an aqueous solution of potassium permanganate, potassium dichromate, or alkali hypochlorite or other oxidising agent, and distilling the product. The two processes may be combined.

T. S. WHEELER.

Purification of [aromatic] hydrocarbons. C. R. DOWNS, Assr. to BARRETT Co. (U.S.P. 1,590,965, 29.6.26. Appl., 7.7.20; cf. U.S.P. 1,318,633, B., 1920, 9 A).—Crude aromatic hydrocarbons, *e.g.*, benzene, toluene, naphthalene, or the like containing impurities such as thiophen and carbon disulphide are mixed in the vapour phase with an equal volume of air and passed at 250—550° for 0·25 sec. over a catalyst consisting of an oxide or a mixture of oxides of vanadium, cobalt, molybdenum, copper, cobalt, manganese, or ruthenium. The impurities are oxidised to sulphur dioxide and carbon dioxide, carbon monoxide, etc., whilst only a small proportion of the hydrocarbon itself is oxidised, and of this the greater part is recovered in the form of useful compounds. Thus from benzene maleic acid and benzoquinone are obtained.

T. S. WHEELER.

Treatment of low-temperature tar phenols [to prevent darkening under the action of light]. Purification of phenols. (A) ZECHE M. STINNES, and A. WEINDEL (G.P. 425,666, 20.11.23), (B) ZECHE M. STINNES, Assees. of A. CORRELL (G.P. 426,344, 29.4.25).—(A) The phenoxide solution from the crude tar distillate is diluted and treated as in G.P. 387,375 (B., 1924, 740) to give a clear liquor. This is then extracted thoroughly with benzene and ether or similar solvents. These together remove the unstable constituents which cause the reddening of the phenols. The extracts are driven off by heating to 180°. They are black solutions, deep red after vacuum distillation. The phenoxide solution is decomposed as usual, and the phenols are distilled *in vacuo*. They form a clear yellow, stable, and pleasant-smelling liquid representing a yield of 68% of the phenols in the tar-oil treated. (B) Crude phenols are suspended in benzene, and stirred with 10% of 86% sulphuric acid at atmospheric temperature or with cooling. The acid is separated and the benzene solution neutralised with ammonia and washed with water. The benzene is then distilled off and the phenols are purified by vacuum distillation. The product is stable to light and without odour.

C. IRWIN.

Purification of phenoxide liquors. ZECHE M. STINNES, Assees. of F. ULRICH (G.P. 426,224, 7.8.24).—Crude phenoxide liquors are subjected to steam distillation under vacuum at 50—60°. Naphthalene, pyridine, and other impurities are driven off without any sensible loss of phenols and the formation of resins is avoided.

C. INWIN.

IV.—DYESTUFFS AND INTERMEDIATES.

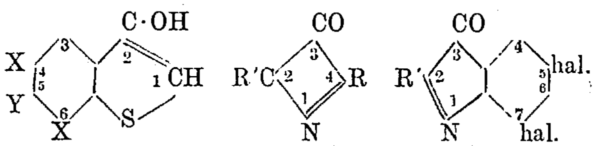
See A., Sept., 949, **Pyrogallol sulphonephthalein, sulphonegallein, 2:3:4-trihydroxybenzoyl-benzene-o-sulphonic acid, and derivatives** (ORNDORFF and FUCHS). 954, **Constitution of catechin. Disintegration products of acacatechin** (NIERENSTEIN). 956, **Synthesis of anthocyanins** (ROBERTSON and ROBINSON); **Diazotisability of aminothiophens** (STEINKOPF and MÜLLER). 960, **Methyl*iso*indigotins and methylindirubius** (WAHL and FAIVRET).

PATENTS.

Manufacture of dyestuffs of the triphenylmethane series. A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 250,785, 29.5.25).—Greenish-blue dyes are made by condensing 1 mol. of *o*-sulphobenzaldehyde with 2 mols. of alkylbenzylarylamines substituted in the benzyl nucleus by halogen and sulpho-groups, and

oxidising the condensation products. The dyes are fast to alkali, and surpass the known similar halogen-free dyes in purity of shade. Examples of suitable alkylbenzylarylamines are the sulphonation products of *o*-, *m*-, and *p*-chlorobenzylethylaniline and 2 : 4- and 2 : 6-dichloroethylaniline. They are made by chlorinating the corresponding chlorotoluenes so as to introduce one atom of chlorine into the side chain, and condensing the products with ethylaniline. m-*Chlorobenzyl chloride*, b.p. 210—215° ; 2 : 4-*dichlorobenzyl chloride*, b.p. 240—243° ; and 2 : 6-dichlorobenzyl chloride, m.p. 39—40°, b.p. 244—245°, are described. A. Davidson.

Violet vat dyestuffs of the 2-thionaphthen-2-indoleindigo series. R. Herz and W. Brunner, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,590,685, 29.6.26. Appl., 11.2.26. Conv., 6.11.24).—2-Hydroxythionaphthen derivatives of the general formula (I)



(I) (II) (III)

where X is halogen or alkyl and Y is H, halogen, or alkyl, are condensed with a 2-isatin derivative of the general formula (II) where R is an aryl group and R′ is halogen or an arylido-group to give dyes formed by doubly linking the 1-carbon of (I) with the 2-carbon of (II). These products dye cotton and wool from an alkaline hyposulphite vat brilliant violet shades fast to light and chlorine surpassing those of known dyes of the same type, and being equal to the best violets of the anthraquinone series in depth and fastness and preferable to them for printing purposes as they are more readily fixed on the fibre and more easily discharged to a pure white. Of especial value are the dyes obtained when X in (I) is halogen, and the isatin derivative has the formula (III). These latter dyes may also be obtained by condensing (I) (X = halogen) with a non-halogenated 2-isatin derivative (R in II = *o*-phenylene) and halogenating the product. The hydroxythionaphthen derivatives (I), all of which are new, are obtained by transforming the corresponding arylamines into the corresponding arylthioglycollic acids and closing the ring either by treatment with an acid condensing agent such as chlorosulphonic acid in the cold, or alternatively converting the arylthioglycollic acid into the corresponding acid chloride and treating this compound with anhydrous aluminium chloride, phosphorus pentoxide, etc. For example, 2 : 4-dichloroaniline is converted into 2 : 4-dichlorophenylthioglycollic acid which yields the corresponding chloride with phosphorus trichloride or thionyl chloride, and this with anhydrous aluminium chloride or phosphorus pentoxide gives 4 : 6-*dichloro-2-hydroxythionaphthen*, m.p. 140—143°. 4 : 5 : 6-*Trichloro-2-hydroxythionaphthen*, m.p. 150—153°, 4-*chloro-2-hydroxy-6-methylthionaphthen*, m.p. 106—108°, 6-*chloro-2-hydroxy-4-methylthionaphthen*, m.p. 120—123°, and 4 : 6-*dichloro-2-hydroxy-5-methylthionaphthen*, m.p. 110°, are also described. As an example of dye formation 5 : 7-dibromoisatin is transformed into 5 : 7-dibromo-2-isatin chloride by treatment with phosphorus pentachloride in chlorobenzene suspension, and the solution is treated with 4 : 6-dichloro-2-hydroxythionaphthen in chlorobenzene at 65°, when the corresponding dye separates. It dyes cotton and wool fast blue-violet shades. 4-Chloro-2-hydroxy-6-methylthionaphthen and 5 : 7-dibromo-2-isatin chloride yield a product which gives fast reddish-violet shades on wool and cotton. T. S. Wheeler.

Manufacture of azo dyestuffs. A.-G. für Anilin-Fabr. (E.P. 244,782, 16.12.25. Conv., 16.12.24).—Dyes of very good fastness to fulling and light are obtained by coupling tetrazotised chloro-substituted 4 : 4′-diaminodiphenylmethanes or their homologues with azo-components. The first components are obtained by condensing chloroanilines or chlorotoluidines with formaldehyde and converting the methylene compounds into diaminodiarylmethanes by the action of a salt of the amine used in presence of acid. Examples are given of wool, direct cotton, and lake dyes prepared from these components. A. Davidson.

Manufacture of yellow azo dyestuffs. I. G. Farbenind. A.-G., Assees. of A. Dorrer (U.S.P. 1,590,728, 29.6.26. Appl., 4.1.26. Conv., 25.2.24).—A diazotised *p*-chloroanilinesulphonic acid, *e.g.*, *p*-chloroaniline-*m*-sulphonic acid, is coupled with diphenylamine and the product, preferably after conversion into the nitroso-derivative, is nitrated with mixed acid to give various nitro-derivatives, which have a great affinity for animal fibres such as silk, leather, and wool, which they dye in fast yellow shades. T. S. Wheeler.

Black disazo cotton dye. Chem. Fabr. Griesheim-Elektron, Assees. of A. L. Laska and A. Zitscher (U.S.P. 1,592,604, 13.7.26. Appl., 2.4.24. Conv., 17.4.23 ; cf. U.S.P. 1,452,083, B., 1923, 647 a).—Diazotised unsulphonated 4 - arylazo - 1 - naphthylamine derivatives containing basic groups in one or several side chains in the aryl or naphthyl nucleus or both are coupled with an arylide of 2 : 3-hydroxynaphthoic acid to yield dyes of the general formula, $(S_n)Aryl\cdot N_2\cdot X(S_n)\cdot N_2\cdot C_{10}H_5(OH)\cdot CO\cdot NHY$, where S is a group of the type $CH_2\cdot NH_2$, $OCH_2\cdot NH_2$, or $O\cdot C_6H_4\cdot NH_2$, X is the residue of an unsulphonated 1 : 4-naphthylene compound, and Y is an aryl group. These products when produced on the fibre dye cotton fast black shades, and when mixed with the usual substrata such as barium sulphate or alumina give black colour lakes. As an example, cotton is dipped in an alkaline solution of the β-naphthylide of 2 : 3-hydroxynaphthoic acid containing Turkey-red oil, and is then immersed in a bath of the diazotised aminoazo-compound from diazotised 4-aminobenzyldimethylamine and α-naphthylamine. A blue-black shade results. T. S. Wheeler.

Reduction of azo compounds to hydrazo compounds. O. W. Brown and C. O. Henke, Assrs. to Newport Co. (U.S.P. 1,589,936, 22.6.26. Appl., 2.11.25).—Aromatic azo- and azoxy-compounds are reduced in good yield to the corresponding hydrazo-compounds by treatment in an organic solvent with hydrogen under pressure in presence of a nickel catalyst. For example,

by treating azobenzene in alcoholic solution at 100° with hydrogen under a pressure of 16·7 atm. in presence of reduced nickel, hydrazobenzene is obtained in satisfactory yield. At 126° and 33·3 atm. aniline is formed in quantitative yield. T. S. WHEELER.

Preparation of condensation products of phenols or phenol ethers and azo-compounds. R. PUMMERER (G.P. 424,863, 4.10.22).—The mixture of the components is heated with acid condensing agents, such as anhydrous zinc chloride, or with free mineral acids, such as hydrogen chloride, or with both. The main products are compounds of undetermined constitution, which are partly soluble and partly insoluble in alkalis. They are either dyes or may be used as intermediates for these. A. DAVIDSON.

Manufacture of intermediates [monoacylated diamines of the anthraquinone series]. BRIT. DYESTUFFS CORP., LTD., W. H. PERKIN, and H. M. BUNBURY (E.P. 255,630, 8.7.25).—Benzoic anhydride or an anhydride of a substituted benzoic acid is used as acylating agent. For example, 100 pts. of 1 : 5-diaminoanthraquinone are stirred with 1000 pts. of nitrobenzene at 175—180° for ½ hr. 110 pts. of benzoic anhydride dissolved in 100 pts. of warm nitrobenzene are added, and the whole is stirred at 175° for 15—20 min. The mixture is cooled to 120° and the precipitated monobenzoyl compound filtered off. A. DAVIDSON.

Making phthalic anhydride. H. D. GIBBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,591,619, 6.7.26. Appl., 30.10.20).—By passing methyl- or other alkyl-naphthalene mixed with about twice the quantity of air required to furnish the necessary oxygen over vanadium or molybdenum oxide at about 400°, phthalic anhydride is obtained. T. S. WHEELER.

Benzidine flakes. W. L. SPALDING, Assr. to NAT. ANILINE & CHEMICAL Co. (U.S.P. 1,591,688, 6.7.26. Appl., 18.6.23).—A rotating cylinder cooled internally by water is partially immersed in molten benzidine and the layer of solid formed on the cylinder is removed by a scraper. The flakes obtained are more readily handled than the lump form hitherto in use. T. S. WHEELER.

Purification of anthraquinone. H. F. LEWIS, Assr. to NAT. ANILINE & CHEMICAL Co. (U.S.P. 1,591,712, 6.7.26. Appl., 4.3.20).—Crude anthraquinone from the oxidation of anthracene with chromic–sulphuric acid is extracted with 5—10 pts. of 5% caustic soda solution at 100° for 1 hr., when anthraquinone of good purity remains. Other alkalis, *e.g.*, potassium hydroxide and barium hydroxide, may also be used. T. S. WHEELER.

Method of sublimation [of benzanthrone]. C. A. LYFORD, Assr. to NAT. ANILINE & CHEMICAL Co. (U.S.P. 1,591,715, 6.7.26. Appl., 2.4.20).—Crude benzanthrone is placed on a perforated plate and superheated steam is passed through it. The benzanthrone sublimes in the current of steam at 100—260°, and is obtained in a relatively pure condition. T. S. WHEELER.

Manufacture of 2-hydroxynaphthalene-3-carboxylic acid-*m*-nitroanilide. CHEM. FABR. ROHNER A.-G. PRATTELN (Swiss P. 111,922, 19.9.24. Addn. to 108,072). —Equimolecular proportions of *m*-nitroaniline and 2-hydroxynaphthalene-3-carboxylic acid are heated in a solution of a tertiary base with phosphorus trichloride. For example, the acid and base are heated at 100° with dimethylaniline until dissolved, phosphorus trichloride is then added slowly, and the mixture stirred for some time longer at 100°. Cold dilute hydrochloric acid is then poured in, the dissolved dimethylaniline hydrochloride removed by filtration, the residue stirred with water, made alkaline with sodium carbonate, and heated until the last traces of dimethylaniline are removed. Technically pure hydroxynaphthoic acid-*m*-nitroanilide is obtained. (Cf. B., 1926, 185.) C. T. GIMINGHAM.

Separation of ortho-chlorotoluene and para-chlorotoluene. A. R. WAHL, Assr. to SOC. ANON. DES MATIÈRES COLORANTES ET PROD. CHIM. DE ST. DENIS (U.S.P. 1,596,325, 17.8.26. Appl., 23.2.21. Conv., 6.3.20).—See E.P. 159,837; B., 1922, 287 A.

Oxazine dyestuff of the anthraquinone series. R. E. SCHMIDT and B. STEIN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,596,460, 17.8.26. Appl., 11.2.25. Conv., 18.2.24).—See E.P. 229,668; B. 1925, 875.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of light on cotton. H. KAUFFMANN (Textilber., 1926, 7, 617—618; cf. B., 1926, 267).—The production of oxycellulose in cellulose during exposure to light is preceded by the formation of "photocellulose," a substance having similar properties (yellow coloration with caustic soda or phenylhydrazine, and reducing action towards silver salts or Fehling's solution) to oxycellulose. The increased affinity of exposed cotton for methylene blue is not directly due to the presence of photocellulose. Cotton is not deleteriously affected by visible light but only by ultra-violet light such as that emitted by a mercury vapour lamp (wave-length about 350 $\mu\mu$). The action of light on cotton is independent of the presence of oxygen (external or occluded) or moisture, and since there is no accompanying change in weight, the resulting deterioration or formation of photocellulose is not the result of oxidation. Photocellulose has the formula $C_6H_{10}O_5$ and its formation in exposed cotton may be followed by an adaptation of the method previously described using potassium permanganate (*loc. cit.*). In bleaching cotton by exposure to sunlight in meadows, the natural yellow to brown pigment is destroyed by visible light, but the cellulose is simultaneously affected by the ultra-violet light, 1—2% of photocellulose being found in cotton fabric thus exposed for 45 days. The deterioration of cotton by light may be prevented by the presence of suitable light-absorbing pigments, but Anthracene Yellow GC and Chrysoidine have been found unsatisfactory for this purpose. A. J. HALL.

Bacterial decomposition of textile fibres. IV. Action of *B. subtilis* and *B. mesentericus* on cellulose. A. C. THAYSEN and H. J. BUNKER (Biochem. J., 1926, **20**, 692—694).—The observations of Trotman and

Sutton (J.S.C.I., 1924, 190 T) that these organisms are capable of decomposing cellulose could not be confirmed.
S. S. ZILVA.

Digestion of spruce wood with dilute nitric acid. O. ROUTALA and J. SEVON (Cellulosechem., 1926, 7, 113—118).—The oxidation of the lignin with 10% nitric acid sets in with a vigorous evolution of gas at about 75—80° and continues for about 15 hrs. in an open vessel at 95°. As the lignin products do not go into solution the penetration of the acid is slow, and well-digested cellulose is obtained only when uniformly and very finely ground material is used. The penetration is greatly assisted by boiling first with aqueous potassium nitrate and adding later the equivalent quantity of sulphuric acid. The yield and quality of the cellulose, after subsequent boiling out with alkali, are approximately comparable with those obtained by the sulphite process of digestion. The necessary proportion of nitric acid is 35—40% of the weight of the air-dry wood, but the actual consumption of nitric acid is about 30%. The reduction products of the nitric acid comprise nitric oxide, ammonia, and nitrogen; a sensible quantity of hydrocyanic acid is also formed, sufficient to make the process dangerous on a large scale. Nitric oxide and ammonia account for 15% of the total nitrogen consumed, but they are produced in too dilute a condition for practical utilisation. The reaction may be expressed as an oxidation of the side chain of the coniferyl complex of the lignin and the nitration of the aromatic nucleus. The latter process comes to an equilibrium with the production of hydrocyanic acid or nitriles.
J. F. BRIGGS.

Rôle of chemistry in the manufacture of silk. W. M. SCOTT (Ind. Eng. Chem., 1926, **18**, 924—926).

Fifty years' progress in the pulp industry. J. D. RUE (Ing. Eng. Chem., 1926, **18**, 917—919).

See also A., Sept., 942, **Constitution of cellulose** (PRINGSHEIM and others); **Soluble cellulose esters of higher fatty acids** (GAULT and EHRMANN). 983, **Hemicelluloses of beech wood** (O'DWYER).

PATENTS.

Conversion of cellular or fibrous [cellulosic] material. E. TAYLOR and E. F. CHANDLER, Assrs. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,592—3, 29.6.26. Appl., [A], 29.7.19. [B], 2.12.19).—(A) The patent relates to the formation of products with an unorganised structure, from cellular organic material. Hydrated copper oxide is added to concentrated ammonia to saturation; cotton is then introduced till it ceases to dissolve. The mass is placed in a closed electrolytic cell provided with a copper anode between two copper cathodes, and an exit tube for ammonia gas. On passing a direct current, cellulose, probably as the hydrate, is deposited in colourless flakes of rubber-like consistency on the anode and copper on the cathodes; ammonia escapes at the top and is recovered for further use. The cellulose obtained is pure, easily soluble in cuprammonia solution, and capable of nitration. (B) The cuprammonia solution of cellulose is fed through hoppers on to a flexible moving copper belt which passes over pulleys through an electrolytic cell containing dilute sulphuric or nitric acid. Four copper plates serve as cathodes, while the belt itself acts as anode. The cellulose is deposited on one side of the belt, copper being deposited on the cathodes. On leaving the cell the belt passes over pulleys into a washing chamber containing water, then into a drying chamber, where the cellulose shrinks from the belt, next into amyl acetate, which improves the appearance and increases the density. Finally, the finished product is wound on rollers. Means are provided for concentrating the cuprammoniacal solution of cellulose before it is fed to the belt, for collecting the ammonia generated in the cell, and for recovering amyl acetate.
R. B. CLARKE.

Treatment of fibrous or cellular [cellulosic] organic material. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,594, 29.6.26. Appl., 11.3.20. Renewed 17.6.24).—The patent relates to an electrolytic method for obtaining cellulose in a definite form, *e.g.*, rods, tubes, filaments etc., from a solution of cellulose in cuprammonia or zinc chloride. The apparatus consists of three closed tanks arranged vertically one above the other, the top tank containing a solution of cellulose in cuprammonia, and the lower tanks very dilute sulphuric acid. The two lower tanks are connected by four tubes, and acid is pumped from the lowest tank, which is also an electrolytic cell, into the middle tank, in order to maintain a head of acid above the tubes. Four smaller tubes, tapering to fine orifices, emerge from the bottom of the top tank and extend downwards into the tubes covered by acid. The cellulose solution emerging from the orifices meets a downward stream of acid which serves to draw out the cellulose as a thread. In the bottom tank these threads fall on to a moving copper belt, immersed in acid, which acts as the anode of the cell. Above the belt is placed a copper cathode. On electrolysis copper is deposited at the cathode and cellulose on the belt. The precipitated cellulose threads are carried to another belt at right angles, washed, and wound on rollers.
R. B. CLARKE.

Production of colloidal material [cellulose]. E. TAYLOR, E. F. CHANDLER, and T. A. HILL, Assrs. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,596, 29.6.26. Appl., 8.10.20).—A method for obtaining sheets of cellulose by electrolysis of a cuprammonia solution of cellulose. The apparatus consists of two vulcanite tanks connected by a pipe at the bottom and by a U-shaped pipe near the top. A pump situated in the U-tube circulates a solution of ammonium nitrate from the right-hand to the left-hand tank, both tanks being completely filled. A smaller vulcanite container, having a felt bottom, fits inside the right-hand tank and rests on a non-conducting flange. Copper plate electrodes are arranged at the top and bottom of each tank, the top plates serving as anodes. The anode of the right-hand tank is arranged just above the container, which is charged with a concentrated cuprammonia solution of cellulose. When a current is passed copper is deposited on the cathodes and a pure film of cellulose is left above the felt.
R. B. CLARKE.

Treating silk and cellulose material. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,600, 29.6.26. Appl., 17.6.24).—A mixture of silk and cotton can be separated by treating it with an ammoniacal nickel solution which dissolves the silk only. The cotton is separated by filtration, and the filtrate is electrolysed in the apparatus described in U.S.P. 1,590,594 (*ante*) to recover the silk in a pure form. R. B. CLARKE.

Treatment of organic material [cellulose]. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,601, 29.6.26. Appl., 17.6.24).—A solution of cellulose in cuprammonia contained in a tank is forced out at the bottom through a nozzle by means of gas pressure. As it emerges it is sprayed, by means of a jet of compressed air, and deposited in finely-divided particles on a moving copper belt which forms the anode of an electrolytic cell. A slow stream of dilute acid falling down into the cell in the same direction as the moving belt prevents the particles of solution from coagulating. The belt on reaching the cell travels above a copper cathode at the bottom and the solvent constituents of the cellulose solution are separated and the cellulose is precipitated. On leaving the cell the particles of precipitated cellulose are removed from the belt by means of a water spray and fall to the bottom of a water tank, whence they are removed and dried. The product consists of very finely divided cellulose.

R. B. CLARKE.

Treating organic material [cellulose] and apparatus therefor. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,606, 29.6.26. Appl., 21.6.24).—In a process for converting fibrous cellulose, *e.g.*, old rags etc., into films, the same apparatus, with modifications, is used for dissolving the cellulose and for electrolytically depositing it in a colloidal form. A tank having a porous screen near its base is filled with dilute aqueous ammonia or water and cellulose is placed on the screen; at the top is an outlet tube connected to a cooling worm, an inlet tube for ammonia gas being placed in between. The worm is connected to a cell filled with ammonia solution and having six copper electrodes. The solution is circulated through the apparatus and ammonia gas is passed in under pressure. When an alternating current is applied a solution of cuprammonia is formed which dissolves the cellulose. When a sufficiently high concentration is reached electrodes of antimony–lead are placed in between the copper plates, and on passing a direct current cellulose is deposited on them. R. B. CLARKE.

Treatment of organic material [cellulose]. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,607, 29.6.26. Appl., 2.7.24).—A film of a cuprammonia solution of cellulose is picked up by a slowly rotating drum around which hot air travels in the same direction, thereby stiffening the film. As it leaves the drum the film passes through dilute acid which increases its strength. It then passes into an electrolytic cell, where the solvent constituents are abstracted. On leaving the cell the material is washed, and to make it non-hygroscopic and suitable for artificial leather it is treated with 30% glycerin and water and dried.

R. B. CLARKE.

Electrolytic cell for deposition of cellulose (U.S.P. 1,590,604 and 1,590,609).—See XI.

Production of artificial leather (U.S.P. 1,590,602). —See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Technical analysis of Indanthrene dye vats. G. DURST and H. ROTH (Z. angew. Chem., 1926, **39**, 900—903).—Simple titration of the vat with mild oxidising agents (*e.g.*, Methylene Blue or ferric ammonium sulphate) does not give the true excess of hyposulphite present, but the total reducing power of the hyposulphite and dye together. By adding formaldehyde to the vat the hyposulphite is stabilised, whilst the dye acids are precipitated, so that, after addition of acetic acid, filtration, and addition of sodium acetate and salicylic acid to the filtrate, the hyposulphite may be determined by titration with ferric ammonium sulphate. The content of dye in the vat may be determined gravimetrically by oxidising the vat with a mixture of ferric ammonium sulphate, sodium acetate, and acetic acid, filtering through a Jena sintered glass crucible, and washing the precipitate thoroughly with acid, alkali, and water. The free sodium hydroxide in the vat is best determined by adding barium chloride (to precipitate sulphites and carbonates) and neutral formaldehyde (to bind the hyposulphite), and then, immediately and without filtering, titrating with $0 \cdot 1N$-acetic acid, with phenolphthalein as indicator.

W. T. K. BRAUNHOLTZ.

Influence of additions to the vat in dyeing with indigo. R. HALLER (Textilber., 1926, **7**, 612—615).—The process of vatting indigo with a hyposulphite and caustic soda is considerably retarded by the presence of protective colloids, particularly glue, gums, gum tragacanth, and soap (Turkey-red oil has less effect). The stability of the vat is decreased, an unusually large number of submicrons are present, the dispersion of the indigo being less, and the resulting dyeings have unsatisfactory fastness to weathering and soap. The presence of methyl, ethyl, and amyl alcohols or acetone assists the dispersion and stability of indigo vats; vats containing methyl or ethyl alcohol contain a very small number of submicrons. Although the resulting dyeings are deeper, their fastness does not appreciably differ from dyeings obtained from a normal vat. Pyridine, and particularly Tetracarnite (Sandoz), increase considerably the dispersion of indigo vats, and the resulting dyeings have greater depth of shade and fastness than those obtained from normal vats of similar concentration. The presence of nitrobenzene assists the vatting of indigo, the resulting vat being orange-red. Indigo vats containing Aktivin (cf. B., 1926, 484) are orange-yellow, and yield dyeings slightly deeper but of normal fastness. Katanol, and wetting agents such as Nekal A and Curacit, salt, and Glauber's salt affect indigo vats unfavourably. The rates of dialysis of indigo vats containing alcohols or acetone, and protective colloids, were rapid and very slow respectively. A. J. HALL.

Use of potassium salts in printing [textiles]. W. SIEBER (Textilber., 1926, 7, 615—616).—The common

practice of replacing potassium salts by the cheaper corresponding sodium salts in printing processes sometimes gives less satisfactory results. White resists for alizarin shades are much clearer when obtained by means of potassium citrate than with sodium citrate, particularly when the resists are over-printed with Aniline Black. A satisfactory resist for the latter process is prepared by adding about 26·3 kg. of potassium hydroxide (free from sodium hydroxide) to 28 kg. of citric acid (crystalline) and 23 litres of water until the mixture is just slightly alkaline, and then adding 18 kg. of gum arabic or gum senegal. In dyeing with ice colours, *e.g.*, Para Red, the progressive discoloration (brown) which occurs with fabric prepared with a solution of β-naphthol in sodium hydroxide may be avoided by use of potassium hydroxide. The dry prepared fabric may then be stored for several weeks without decomposition or discoloration, and the resulting shade in the case of Para Red is bluer than that similarly obtained with sodium hydroxide. A prepare made with potassium hydroxide is especially suitable for the production of Para Red discharges on Aniline Black. A. J. HALL.

[Preparation of] discharge printing pastes containing caustic soda and having no deleterious action on [copper] printing rollers. W. SIEBER (Textilber., 1925, **6**, 829—830; 1926, **7**, 244).—Printing pastes suitable for discharging fabric mordanted with tannic acid are prepared by adding caustic soda to a solution of gum arabic or senegal, it being particularly essential to use efficient stirring and cooling so as to avoid coagulation of the gum. For example, 53·5 kg. of caustic soda of 36° B. (*d* 1·334) are added slowly to 20 litres of a 50% solution of gum senegal, the temperature being strictly maintained at 20—22°. A more effective discharge paste is obtained by incorporating 1700 g. of potassium hydroxide in the paste described above. The familiar damage caused to printing rollers by use of similar pastes prepared, with heating or without cooling, with starches and British gum, is due to the adherence of glutinous lumps of the paste to the printing rollers and doctors; it is not due to the separation of crystalline sodium salts. A. J. HALL.

Textile dyeing [during the past fifty years]. W. K. ROBBINS (Ind. Eng. Chem., 1926, **18**, 927—929).

PATENTS.

Treatment of textile fibres, yarns, fabrics, or the like for obtaining effects of colour or lustre, or both. N. V. NEDERLANDSCHE KUNSTZIJDEFABRIEK (E.P. 245,407, 7.5.25. Conv., 30.12.24).—See F.P. 597,231; B. 1926, 437.

Multi-chamber yarn dyeing apparatus. P. F. HÖLTZING (E.P. 256,854, 15.1.26).

Yarn-dyeing apparatus. P. F. HÖLTZING (E.P. 257,160, 15.1.26).

Black disazo cotton dye (U.S.P. 1,592,604).—See IV.

Dyeing of leather (E.P. 255,555).—See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Catalytic removal of oxygen from gas mixtures containing hydrogen [*e.g.*, ammonia-synthesis gases]. J. A. ALMQUIST and E. D. CRITTENDEN (Ind. Eng. Chem., 1926, **18**, 866—867).—Copper prepared by the reduction of copper oxide is more effective than massive copper in promoting the catalytic removal of oxygen as water from, *e.g.*, by-product nitrogen to be used in the synthesis of ammonia. Copper in any form is a more suitable catalyst than platinum. B. W. CLARKE.

Determination of water-soluble barium in black ash. W. F. MEISTER and T. STEPHENS (Ind. Eng. Chem., 1926, **18**, 828—829).—The water-soluble barium in barium black ash may be accurately determined by boiling 0·2 g. of the ash with 125 c.c. of water for 5 min., filtering rapidly through asbestos, washing the residue with 100 c.c. of boiling water, and boiling the filtrate and washings with 25 c.c. of 0·5*N*-sulphuric acid for 5 min. After settling for 1 hr. the precipitated barium sulphate is collected, ignited, and weighed. A rapid volumetric control method may be carried out on 2 g. of ash by treating the filtrate with 50 c.c. of sulphuric acid (23·16 g./litre), boiling until all hydrogen sulphide is expelled, and titrating the excess of acid with sodium hydroxide solution of the same strength, using phenolphthalein as indicator. Addition of a correction of 0·5 c.c. to the volume of sulphuric acid used is necessary, this representing the difference in the end-points for phenolphthalein and methyl-orange; the corrected volume multiplied by 2 gives the percentage of barium sulphide in the sample. J. W. BAKER.

Reagent and standard for borax. L. S. WEATHERBY and H. H. CHESNY (Ind. Eng. Chem., 1926, **18**, 820—821).—Borax, either alone or mixed with other salts, is accurately determined by direct titration with 0·1*N*-hydrochloric acid using methyl-orange as an indicator, in the presence either of mannitol, or the much cheaper commercial dextrose (cerelose). The results obtained using lævulose, glycerol, honey, or invert sugar are less satisfactory. Borax in alkaline brine solutions containing sodium carbonate or sodium hydrogen carbonate may also be thus determined if the alkalinity of the borax and other alkalis is first determined separately, and colour standards then prepared corresponding to the calculated end-points of the mixtures. J. W. BAKER.

Chlorination of metals. P. S. BRALLIER (Trans. Amer. Electrochem. Soc., 1926, **49**, 65—71).—A description of the preparation of some metal chlorides with special reference to large-scale production. Silicon tetrachloride may be prepared by the action of chlorine at a high temperature on a mixture of 90% silicon and silicon carbide. A vertical shaft furnace is advocated consisting of a water-cooled steel or cast-iron shell, lined with carbon slabs, with a thin layer of dry sand between the carbon and steel. The condenser should be flushed with cooled liquid silicon tetrachloride to prevent choking by ferric or aluminium chloride. Titanium tetrachloride may be prepared similarly using titanium carbonitride containing 70—75% of titanium. The heat of formation

of titanium tetrachloride is roughly determined as 156,000 cal. per g.-mol. Tin tetrachloride may be prepared by the action of chlorine on metallic tin immersed in liquid stannic chloride containing a little sulphur. If the raw material is tin-plate, this may be directly treated with chlorine, but with pig tin the rise of temperature is too great and the above method is better. Antimony trichloride may be prepared by the direct action of chlorine on metallic antimony at about 80—90°, but careful regulation is essential to prevent rise of temperature and fusion of the metal. Zinc chloride can be prepared by bubbling chlorine into molten zinc kept at about 500° in a steel or cast-iron box lined with refractory clay. The crude chloride rises to the top and is tapped off; it contains finely-divided zinc and zinc oxide (" blue powder ") in suspension, but a product of 99·8% purity is obtained on redistillation.

W. Hume-Rothery.

Fire risk of concentrated hydrogen peroxide solutions. G. Agde and E. Alberti (Z. angew. Chem., 1926, **39**, 1033—1035).—A series of experiments undertaken to elucidate the cause of a fire in a railway wagon loaded with wicker-cased carboys containing 60% hydrogen peroxide, to which stabiliser had been added, showed that used engine-room woollen waste inflamed spontaneously when brought into contact with the solution at ordinary temperatures. Clean waste, soaked with oil, did not ignite under these conditions, but when iron filings were also present, inflammation followed Sweepings from wooden floors and even fine sand had the same effect as iron filings, causing even fairly thick wood, saturated with the peroxide solution, to ignite. A very small glowing charred patch in wood is sufficient to cause intense combustion when the solution is brought into contact with it.

S. I. Levy.

Production of iodine in Chile. J. B. Faust (Ind. Eng. Chem., 1926, **18**, 808—811).—Average Chilean " caliche " contains 0·15% of iodine in the form of sodium iodate. Owing to the repeated use of mother liquors for leaching the caliche, the iodate accumulates until the liquor contains 6—12 g. of iodine per litre. The extraction of iodine is performed by reduction of iodate with sodium bisulphite. Sodium carbonate is prepared by burning 100 pts. of sodium nitrate with 16—20 pts. of coal, and extracting the cooled mass. The settled liquor (*d* 1·12) is saturated with sulphur dioxide prepared by burning sulphur. The iodate mother liquor is mixed with an excess of bisulphite solution in a wooden tank, nearly neutralised with sodium carbonate, and the quantity of mother liquor necessary to complete the reaction added. The iodine is allowed to settle, washed, and hand-pressed, yielding a product containing 75—80% of iodine. This is broken up and heated over direct fire in cement-lined retorts. The vapours are condensed in earthenware pipes, any water being allowed to drain away from the joints. The regular working efficiency of an average plant will not exceed 65—70%. The chief losses occur in weak liquors run away. Large losses are also usual in preparing the bisulphite solution. The amount of mother liquor which can be treated for iodine is limited by the fact that this treatment means the addition of water to the nitrate cycle and consequently more loss of nitrate in the latter. Other methods which have been suggested for iodine extraction are reduction with sodium thiosulphate and sulphuric acid, and reduction with sulphur dioxide direct. The latter method would reduce the dilution of the liquors, but the sulphuric acid produced would require to be neutralised. The cost of production of iodine under present conditions is estimated at 50—75 cents per kg. Owing to economic circumstances only 2% of the iodine available is at present extracted.

C. Irwin.

Preparation of aqueous thiocyanic acid. Gluud, Keller, and Klempt.—See A., Sept., 943.

Ammonia recovery by gypsum. Jackman.—See II.

Manufacture of sulphuric acid. Metallbank u. Metallurgische Ges. A.-G. (G.P. 427,541, 4.4.25. Addn. to 370,853; cf. E.P. 184,966, B., 1922, 858 a).—In the plant described in the previous patent there was used in the middle section nitrosylsulphuric acid of lower sulphuric acid content than that used in the front and rear sections. According to the present patent an additional acid cycle is introduced which is independent of the rest of the plant. In this section, consisting of several towers in series, nitrosylsulphuric acid of *d* about 1·71 is used as trickling liquid. The gases are led up these towers in turn. This effects a concentration of the acid, which is taken up to the top of the first tower after leaving the last, thus rendering the process continuous.

R. B. Clarke.

Manufacture of ammonium phosphates. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 255,766, 3.3.26).—Phosphoric anhydride is absorbed from gases containing it by ammonium phosphate solution, ammonia in the quantity required for the desired ammonium phosphate being added before, after, or during the absorption. A hot saturated solution is obtained and the solid ammonium phosphate separates on cooling, the mother liquor being used repeatedly for the absorption.

W. G. Carey.

Manufacture of diammonium phosphate. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 256,137, 22.2.26).—Phosphoric acid and ammonia are added to a saturated solution of diammonium phosphate while maintaining a ratio of 1 molecular proportion of acid to between 1·5 and 2 molecular proportions of ammonia, these proportions being controlled by titration, hydrogen-ion concentration measurements, or indicators with suitable transition points. The heat of the reaction evaporates the required amount of water, and the solution is cooled and gaseous ammonia is added until the solution contains 2 proportions of ammonia to 1 of acid, when pure diammonium phosphate separates.

W. G. Carey.

Carrying out exothermic gaseous catalytic reactions [especially the synthesis of ammonia]. Synthetic Ammonia & Nitrates, Ltd., and H. A. Humphrey (E.P. 255,963, 4.5.25).—In a reaction of

the kind mentioned in the title a more even distribution of temperature in the catalytic mass is secured by periodic reversal of the gas flow. C. IRWIN.

Carrying out exothermic gaseous catalytic reactions [especially the synthesis of ammonia]. SYNTHETIC AMMONIA & NITRATES, LTD., and R. E. SLADE (E.P. 255,964, 4.5.25).—A catalyst chamber is constructed narrow at the centre and becoming wider towards the inlet and outlet ends, and is fitted with a number of transverse heat-insulating partitions. On commencing the reaction the centre portion is electrically heated. When reaction is established the applied heat is withdrawn, and the zone of maximum temperature gradually travels forward in the direction of the gas flow. When it nears the end of the chamber the gas flow is reversed and it is caused to travel back again. By this method of working a heat interchanger is rendered unnecessary, the catalyst mass itself performing that function. With intense reaction artificial cooling may be necessary, but otherwise the gaseous products of reaction pass away at about 100° only. C. IRWIN.

Method of forming nitrogen-hydrogen mixtures. J. C. CLANCY, Assr. to NITROGEN CORP. (U.S.P. 1,594,815, 3.8.26. Appl., 18.2.22).—In the production of a nitrogen-hydrogen mixture from atmospheric nitrogen, the heat generated by causing the atmospheric oxygen to unite with hydrogen is used to heat a quantity of metal, over which the gaseous mixture is passed to remove the last traces of oxygen. H. ROYAL-DAWSON.

Tricalcium cyanide. R. W. POINDEXTER, JUN., Assr. to CALIFORNIA CYANIDE CO. (U.S.P. 1,592,127, 13.7.26. Appl., 21.11.25).—By combining calcium oxide, water, and hydrogen cyanide in exactly the required proportions, $Ca_3(OH)_4(CN)_2$ is obtained as a whitish pulverulent compound, dissociating on treatment with water into calcium hydroxide and calcium cyanide, which evolves hydrocyanic acid continuously. The compound is valuable as a source of hydrocyanic acid for fumigating, as it keeps well in the absence of moisture. E. S. KREIS.

Reduction of neutral and acid metal-salt solutions. H. BARDT (U.S.P. 1,592,173, 13.7.26. Appl., 18.11.24. Conv., 7.3.23).—When copper solutions are reduced by treating with cellulosic material, the separation of the metal from the fibrous residue is expensive. According to the present patent the cellulosic material is hydrolysed by heating under pressure with an inorganic acid or salt for several hours, and the resulting clear solution is used for reducing the copper solution. E. S. KREIS.

Prepared metallic chloride and method of making it. H. H. DOW, Assr. to DOW CHEMICAL CO. (U.S.P. 1,592,971, 20.7.26. Appl., 26.9.25).—The patent relates to a method of producing calcium chloride or similar hygroscopic chloride in a form which can be packed without setting and hardening. A cooled rotating drum dipping into molten calcium chloride, corresponding to the composition $CaCl_2,2H_2O$, picks up a thin layer, which is scraped off and conveyed to the top of an inclined rotary cylinder. Here it is mixed with anhydrous powdered calcium chloride, introduced through a hopper. The material passes down the wall of the cylinder, and travels over a screen at the bottom which removes any powder in excess of that required to coat the flakes. R. B. CLARKE.

Flake magnesium chloride and method of making it. W. R. COLLINGS, Assr. to DOW CHEMICAL CO. (U.S.P. 1,593,440, 20.7.26. Appl., 28.11.21).—Magnesium chloride hexahydrate, containing slightly less than the normal amount of water of crystallisation, is prepared by picking up the molten salt at 169° in a thin layer on a revolving drum, from which it is scraped after cooling. The product, which is non-caking, has a glass-like finish, and is much harder than the normal hexahydrate. R. B. CLARKE.

Producing barium chloride. A. JAHL, Assr. to J. MICHAEL & CO. (U.S.P. 1,593,902, 27.7.26. Appl., 6.3.24. Conv., 5.12.22).—A solution containing barium sulphide and barium hydrosulphide is treated first with solid barium chloride and then with an alkali chloride. C. IRWIN.

Products from barium sulphide. RHENANIA VEREIN CHEM. FABR. A.-G., ZWEIGNIEDERLASSUNG MANNHEIM, and F. RÜSBERG (G.P. 426,925, 20.10.23. Addn. to 423,755, B., 1926, 322).—Decomposition of barium sulphide is carried out by means of suitable acids or similarly acting materials in a solution of barium chloride. A. COUSEN.

Making zinc sulphide. F. G. BREYER and C. W. FARBER, Assrs. to NEW JERSEY ZINC CO. (U.S.P. 1,594,001—2, 27.7.26. Appl., 7.4.25).—(A) A mixture of zinc oxide pigment and sulphur is heated to a temperature at which the formation of zinc sulphide takes place readily, and the product is subjected to a heat treatment under non-oxidising conditions. (B) The reaction is carried out by passing the zinc oxide-sulphur mixture progressively through a long vertical reaction chamber maintained at a suitable temperature to allow the reaction to take place readily. A. R. POWELL.

Treating salt solutions. W. H. BINDER (U.S.P. 1,594,707, 3.8.26. Appl., 5.11.25).—Potassium chloride is added to a saturated solution containing potassium chloride, sodium carbonate, sodium chloride, sodium sulphate, and borax; the liquor is heated to dissolve the potassium chloride, and then added to a dry mixture of the salts mentioned, and again heated, whereby the sulphate, carbonate, and chloride of sodium remain undissolved, and are separated from the liquor. On cooling the liquor potassium chloride separates, and on further cooling, the borax crystallises out. H. ROYAL-DAWSON.

Manufacture of alkali metabisulphites or bisulphites. F. LEROY (F.P. 605,115, 29.10.25. Conv., 26.10.25).—A continuous process in which a suspension of a normal sulphite in a saturated solution, obtained by neutralising a solution of bisulphite with solid alkali carbonate or bicarbonate, is saturated with sulphur dioxide. The carbon dioxide generated is recovered. R. B. CLARKE.

Manufacture of arsenic pentoxide. P. Askenasy and E. Elöd (E.P. 255,522, 21.4.25).—150 pts. of arsenious oxide and 150 pts. of 60% nitric acid are introduced into a closed vessel having a stirrer and a jacket. A small quantity of arsenic pentoxide or other catalyst may be added. Oxygen at 20 atm. is then forced in and the mixture stirred for 12—18 hrs. at 70—90°. The nitric acid remains nearly unchanged and can be distilled off, the arsenic acid solution being afterwards converted into calcium arsenate, if desired. Arsenious sulphide may be used in place of the oxide. C. Irwin.

Apparatus for manufacture of carbon disulphide. H. Schulz (G.P. 407,656, 1.4.23).—Sulphur and charcoal are heated in a chamber with internal heating, as, *e.g.*, by means of vertical heating resistances or pipes through which generator gas or the like is passed. The resistances are provided with protective coverings of suitable material, preferably metal, and these are fixed in tubes of firebrick or the like. The walls of the furnace are of refractory material, with inner and outer metal coverings, and arrangements are made for heating and vaporisation of the sulphur in suitable spaces in the furnace walls. The heating is so arranged in relation to the furnace walls that the latter are more strongly heated in places where the sulphur deposits than elsewhere. C. T. Gimingham.

Preparation of concentrated solutions of hydrogen peroxide. E. de Haën A.-G. (G.P. 428,707, 10.4.24).—In the preparation of concentrated hydrogen peroxide solutions from phosphoric acid and barium peroxide, barium carbonate is added to the latter in increasing amounts as the reaction proceeds, until at the conclusion the barium peroxide may even be completely displaced. Other neutral carbonates, as those of calcium, strontium, or magnesium, or the bicarbonates of the alkalis, may be used in place of barium carbonate. A. Cousen.

Separating gas mixtures, more especially air or other difficultly liquefiable gas mixture. R. F. and R. K. E. Mewes (U.S.P. 1,594,336, 27.7.26. Appl., 12.3.24. Conv., 16.2.22).—In the case of a rectifying column fractionating liquid air under high pressure, a surplus of liquid nitrogen is produced at the top and a surplus of oxygen evaporated at the bottom. The descending liquid nitrogen is evaporated and the ascending oxygen gas liquefied, within the column. C. Irwin.

Gas purification [removal of carbon monoxide from ammonia-synthesis gases] by ammoniacal cuprous solutions. J. D. Dely, Assr. to Atmospheric Nitrogen Corp. (U.S.P. 1,597,345, 24.8.26. Appl., 16.8.23).—See E.P. 220,651; B., 1925, 957.

Removal of calcium and magnesium from rock-salt brine. F. A. Freeth and L. A. Munro, Assrs. to Solvay Process Co. (U.S.P. 1,597,370, 24.8.26. Appl., 21.8.20. Conv., 15.2.19).—See E.P. 149,707; B., 1920, 689 A.

Cooling hot salt solutions (G.P. 430,482).—See I.

Manufacture of calcium carbide etc. (F.P. 605,530).—See II.

VIII.—GLASS; CERAMICS.

Properties of fused quartz and other forms of silicon dioxide. H. L. Watson (J. Amer. Ceram. Soc., 1926, 9, 512—534).—A compilation of data on the physical properties of the crystalline modifications of silica, the physical and chemical properties of vitreous silica, and a short account of the cryptocrystalline and hydrated amorphous forms of the oxide. A. Cousen.

Hydrogen-ion measurements on clay slips. D. W. Randolph and A. L. Donnenwirth (J. Amer. Ceram. Soc., 1926, 9, 541—547).—For determination of the p_H value of clay slips (clay 1, water 12) an electrometer of calomel and hydrogen electrodes was used. No change in the p_H value was noted when ball clays and kaolins were aged for 2—3 weeks. A definite relationship was found between the dry strength of a clay and the p_H value of the slip from which it was obtained. The relationship between viscosity and p_H value for any particular clay depended largely on the electrolyte used. The effect of a change of p_H of a ball clay on the plasticity (as measured by Bingham's method) was also determined. A. Cousen.

Physical properties of ceramic bodies. F. Singer (Z. Elektrochem., 1926, 32, 382—395).—Ceramic products are arranged according to their common nomenclature in seven groups. Considered solely from the point of view of chemical composition, these groups are shown to vary from two-component to multi-component systems. All the three- and four-component systems are based on the two-component system Al_2O_3–SiO_2; *e.g.*, in combination with alkalis as porcelain and earthenware; with alkalis and lime as earthenware; and with alkalis and iron oxide as stoneware. Unlike fusions, sintered ceramic bodies are characterised by incomplete physical and chemical homogeneity in the liquid phase; they contain, in addition to a fused glassy matrix, undissolved crystallites of raw material and newly-formed crystals. The latter determine the characteristics of the product. The results of mechanical and thermal tests on the various types of ceramic products are given in tables. Photomicrographs are reproduced to illustrate the importance of the effect of mullite crystals, their arrangement and distribution, upon the physical properties of ceramic bodies. The modern aim is to produce the maximum number of minute mullite crystals in close, felty formation embedded in a homogeneous glass. F. Salt.

Deformation of ceramic messes on drying. O. Krause (Kolloid-Z., 1926, 39, 263—264).—Ceramic masses of high plasticity, especially those rich in kaolinite, show on drying a twisting in a direction opposite to that in which they were worked on the potter's wheel. In some cases this torsion manifests itself by the formation of characteristic cracks during firing. N. H. Hartshorne.

Methods of testing and physical properties of wet-process electrical porcelain. L. Navias (J. Amer. Ceram. Soc., 1926, 9, 501—510).—In the determination of compressive strength of electrical porcelain it is suggested that the ultimate failure should be recorded

as it is more definite and constant than the initial failure. The height of the specimen is an important variable, and a cylindrical specimen, 1 sq. in. in area and $1\frac{1}{8}$ in. high, is recommended. A test-piece of similar area is recommended for the transverse strength. As the area of minimum cross-section increases the value obtained for tensile strength increases rapidly, and the diameter of the test-piece should be given, together with the determined values. A convenient apparatus for determining tensile strength is described.

C. A. King.

Electrical resistance of refractory materials. J. B. Ferguson (Canad. Chem. Met., 1926, **10**, 131—136).—Refractory materials are regarded as crystalline aggregates cemented together by a glassy bond. The results of previous work on the electrical properties of glasses and crystals are correlated on the basis of the Rasch–Henrichsen formula: $\log \rho = A/T + B$, in which ρ is the resistivity, T is the temperature (absolute scale), and A and B are constants peculiar to the substance. By plotting $\log \rho$ against $1/T$ a straight-line relationship where the rule is obeyed is obtained. Changes of slope are observed in certain critical zones. With Royal Berlin porcelain a slight change of slope occurs at about 1070°. Talc follows the rule fairly closely. Slight changes of slope are observed with sillimanite and magnesite. With kaolin and flint fireclay changes occur at the temperatures at which chemical changes take place. The curve obtained with a firebrick suggested continuous changes in the material; silica bricks followed the rule to a high temperature, at which the resistivity decreased suddenly, indicating that actual melting had taken place; with magnesite bricks the rule held over the whole temperature interval. For ordinary commercial refractories the plotted results may be expected to fall on a series of straight lines, changes of slope occurring at the melting interval of the matrix, at the inversion temperatures of the crystals present, and at the point where chemical reaction takes place.

F. Salt.

Firing terra-cotta in an open kiln. O. E. Mathiasen (J. Amer. Ceram. Soc., 1926, **9**, 548—550).—Terra-cotta was successfully fired in open oil-fired kilns of the regular rectangular type, but having special flues and combustion chamber. Details of the flues and combustion chamber and method of working are given.

A. Cousen.

Fifty years of glass-making. A. Silverman (Ind. Eng. Chem., 1926, **18**, 896—899).

A half-century of progress in the glass industry. G. W. Morey (Ind. Eng. Chem., 1926, **18**, 943—945).

Influence of chemistry on ceramics [during the past fifty years]. R. C. Purdy (Ind. Eng. Chem., 1926, **18**, 952—953).

Production from raw clay of material stable towards water. Budnikov.—See IX.

Inside frosting of electric lamps. Pipkin.—See XI.

Patents.

Fining glass. Soc. Anon. des Manuf. des Glaces et Prod. Chim. de St. Gobain, Chauny et Cirey (E.P. 250,536, 5.2.26. Conv., 10.4.25).—Glass is fined by causing it to flow from the lower region of the melting chamber, as a thin sheet, into the first portion of the fining chamber, which possesses a raised hearth, the glass being subjected completely to an elevated temperature therein. The second part of the fining chamber is deeper and at a lower temperature than the first, and the glass flows down a slope to the bottom of this zone, finally passing out from below into the pouring zone. Modifications of the above: (1) the fining zone has a second portion without slope; (2) the connecting channels between the melting and fining zones are heated electrically; (3) suitable heating and cooling arrangements are provided to maintain the desired temperatures in the various zones.

A. Cousen.

[Alloy for] glass-melting container. J. Bryce (U.S.P. 1,592,336, 13.7.26. Appl., 30.1.23).—An alloy containing Fe (73—53%), Cr (25—45%), Si, C etc., is used to construct containers for the manufacture of glass; the alloy is completely resistant at all the temperatures employed to the action of the ingredients forming the batch.

T. S. Wheeler.

Treating glass. C. A. Kraus (U.S.P. 1,592,429, 13.7.26. Appl., 5.6.24).—A glass surface is treated with silver nitrate solution and heated at 160° for 10 min. It is then cooled, washed, and reheated at 220° for 20—60 min. Some of the sodium ions present in the surface of the glass are replaced by silver ions, and although the presence of the silver ions is normally invisible, yet on causing liquid to condense on the glass, *e.g.*, by breathing on it, the effect is apparent. The method is suitable for trade-marking glass articles, such as lenses.

T. S. Wheeler.

Melting of quartz glass and other highly refractory materials. Deutsch-Engliche Quarzschmelzes G.m.b.H., and Z. von Hirschberg (G. P. 428,654, 28.2.24).—As a fuel, the vapour of a liquid hydrocarbon such as benzene, benzine, petroleum, etc., or mixtures of these, is mixed with oxygen under pressure. Such a fuel is more convenient than acetylene and allows of better observation of the material.

A. Cousen.

Manufacture of pottery. R. G. Varcoe (E.P. 255,607, 16.6.25).—As a source of silica and alumina in a cheap, rapidly mixed form for potter's clay, china clay waste or tailings is dried and powdered. Approximately 76% of this is incorporated with 14% of ball clay, 5% of china clay, $2\frac{1}{2}$% of flint, and $2\frac{1}{2}$% of china stone.

A. Cousen.

Manufacture of porcelain bodies which can be easily fused and cast. H. Becker, sen. (G.P. 430,387, 1.4.25).—Fluorspar is smelted with glass waste, or a similar material, containing a small quantity of alumina which decreases the volatilisation of silicon fluoride and, consequently, damage to the furnace walls; the quantity of fluorspar should be at least 20% of the total. If a metal oxide be added to the melt a metallic lustre is imparted to the surface of the finished product.

R. B. Clarke.

Fire- and acid-proof plastic substances [refractories]. A. Wolfsholz (E.P. 256,790, 24.8.25).—See F.P. 602,475; B., 1926, 632.

IX.—BUILDING MATERIALS.

Action of sodium and magnesium sulphates on Portland cement. G. R. Shelton (Ind. Eng. Chem., 1926, **18**, 854—856).—A suspension of hydrated commercial Portland cement in water contained particles of gel, hexagonal plates of hydrated tricalcium aluminate, crystals of hydrated lime, and sulphoaluminate crystals. On treatment with sodium and magnesium sulphate solutions, the crystals of lime and tricalcium aluminate rapidly disappeared, leaving amorphous masses of gel which subsequently became granular. The sulphoaluminate crystals remained unaltered and were probably formed from the gypsum present in the cement. A white Portland cement containing tricalcium aluminate, tricalcium silicate, and β-dicalcium silicate yielded different results, no sulphoaluminate being found while the crystalline grains in the suspension persisted considerably longer. In both cases the disintegration was more rapid than with pure hydrated tricalcium aluminate (cf. B., 1926, 91), probably owing to the presence of silicates. The action of sulphate solutions on cement clinker was also determined.

B. W. Clarke.

Action of sulphate water on concrete. D. G. Mills (Public Roads, 1925, **6**, 174—179, 183; Chem. Abstr., 1926, **20**, 2056).—Cylinders of high-alumina cement and of standard cement cured in steam at 100° were unchanged, whereas cylinders of standard cement cured in water vapour at lower temperatures deteriorated, after immersion for 1 year in a lake water containing 2·34—4·72% of salts, chiefly magnesium and sodium sulphates.

A. A. Eldridge.

Effect of calcium chloride on strength of concrete. A. S. Levens (Eng. News-Rec., 1926, **97**, 214—215).—The addition of 2% of calcium chloride slightly increases the strength of concrete, higher percentages resulting in a decrease of strength. The texture of the concrete is also improved, but the shrinkage is increased considerably, which renders the use of calcium chloride dangerous in concrete structures. The addition of calcium chloride, however, enables a high strength to be developed when curing in dry air.

B. W. Clarke.

Production from raw clay of material stable towards water. P. P. Budnikov (Kolloid-Z., 1926, **39**, 269—275).—With the object of producing a cheap building material experiments have been carried out on the stabilising action towards water of various substances on raw clay. The addition of alkalis up to about 5% increases the susceptibility to attack by water, larger additions make the clay immune, whilst with still larger quantities the clay is again attacked by water. Phosphoric acid (3—6%), free or in the form of a mixture of tricalcium phosphate, superphosphate, or phosphorite and sulphuric acid, renders the clay quite resistant to attack. It also reduces the shrinkage on drying, particularly if 10% of asbestos be added. It has no marked effect on the tensile strength except when added as calcium phosphate and sulphuric acid, when it increases it by 50%. Unfortunately, the action of phosphoric acid depends on the composition of the clay; *e.g.*, in a series of clays having a silica content ranging from 5·5 to 42·5%, only those with 27·0% and under were stabilised completely by the above amount, viz., 3—6%. Experiments designed to explain the action of phosphoric acid were inconclusive. It probably depends on the colloidal nature of the clay and the coagulative action of the acid.

N. H. Hartshorne.

Portland cement industry [during the past fifty years]. R. K. Meade (Ind. Eng. Chem., 1926, **18**, 910—913).

Patents.

Burning material [cement] in rotary kilns. M. Vogel-Jorgensen (E.P. 255,569, 4.5.25).—Slurry, *e.g.*, cement slurry, is dried in the kiln in the usual way to form nodules, which are ground before reaching the decarbonating zone by means of grinding members in the interior of the kiln or in a grinding chamber communicating with the kiln and arranged to prevent the leakage of air into the kiln. The grinding process facilitates the expulsion of carbon dioxide from the material and increases the capacity of the kiln.

B. W. Clarke.

Improving the properties of articles formed of fibrous materials with cement binding agents. W. H. Kobbé, Assr. to Texas Gulf Sulphur Co. (U.S.P. 1,594,417, 3.8.26. Appl., 22.5.24).—The articles, *e.g.*, asbestos-cement products, are impregnated wholly or in part with sulphur. (Cf. B., 1926, 668.)

H. Royal-Dawson.

Cement manufacture. E. G. Weeks, Assr. to Merz & McLellan (U.S.P. 1,596,956, 24.8.26. Appl., 7.2.22. Conv., 17.3.21).—See E.P. 181,811; B., 1922, 635 a.

Cold bituminous paving composition. O. H. Berger (E.P. 257,114, 28.9.25).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Elimination of sulphur from spathic iron ore by roasting. J. Ruhrmann (Stahl u. Eisen, 1926, **46**, 1118—1119).—In order to test on a large scale the results obtained by Weyel (cf. B., 1925, 762) on a laboratory scale several tons of a spathic iron ore were roasted in a cylindrical Siegerland furnace and the sulphur content of the product determined at different heights in the furnace. In the upper three-fifths of the furnace 31% of the total sulphur was removed and in the lower two-fifths a further 21%. Passing steam through the furnace did not have any appreciable effect, but quenching the red hot charge in water reduced the sulphur content by 24—34%. Prolonged leaching of the roasted ore with water removed 60% of the sulphur content, but only a small fraction of the manganese, so that it seems that the greater part of the sulphur remaining after roasting is in the form of ferrous sulphate.

A. R. Powell.

Factors influencing the rate of pickling of sheet iron. J. E. Hansen and G. S. Lindsey (J. Amer. Ceram. Soc., 1926, **9**, 481—492).—A freshly made sulphuric acid (6%) pickling bath was found to clean iron more quickly than one in which ferrous sulphate was

present, and there was no evidence that the addition of a portion of old pickling solution improved the rate, contrary to general practical experience. Ferric sulphate accelerated the rate of pickling, but was converted quickly into the ferrous salt with a retarding effect. Alteration in concentration of acid influenced the speed of reaction, but not to the same degree as variation in temperature of the solution, and the addition of hydrochloric acid or sodium chloride to the solution retarded the process. Contrary to theoretical views, increased concentration of ferrous chloride in a 11·45% hydrochloric acid bath at 30° accelerated pickling. Annealed iron lost 250 to 400% more in weight during the early stages of treatment than unannealed iron, and the process was not so easily controlled. Contact of iron with a containing basket of Monel metal increases the pickling rate by reason of the electrochemical effect.

C. A. KING.

Is the direct change from austenite to troostite possible? K. HONDA (Iron and Steel Inst., Aug., 1926. Advance proof, 4 pp.).—From a consideration of the crystal structure of austenite and martensite and the mechanism of the Ar1 transformation it is concluded that troostite is invariably the decomposition product of martensite, and not a product formed by direct decomposition of austenite. The direct change from austenite to troostite is not possible. The carbon in solution in austenite and martensite is present as carbon atoms, and not as cementite molecules.

M. COOK.

Spheroidising of cementite. B. STOUGHTON and R. D. BILLINGER (Ind. Eng. Chem., 1926, **18**, 785—788). —The Ac1 points of six specimens of steel, viz., file steel (hypereutectoid) carbon 1·4%, eutectoid carbon 0·80%, and hypereutectoid, carbon 0·45%, were determined, and the steels heated for varying periods in the neighbourhood of these points, both below and above. Brinell hardness tests taken before and after treatment showed a considerable softening in all cases (even below the Ac1 point). Spheroidisation took place on each occasion, and may be accomplished within a temperature range extending from about 30° below the critical point to 70° above this temperature. Contrary to Honda and Saito (B., 1921, 12 A), spheroidisation of lamellar pearlite can take place below the Ac1 point. The micrographic results appear to indicate that there is actual spheroidisation of pearlitic cementite and not simply a breaking up of grain boundaries. Spheroidisation is associated with the softest and weakest, but most ductile state of a steel.

A. COULTHARD.

Carburisation and decarburisation of iron, and surface decarburisation of steel. A. JOHANSSON and R. VON SETH (Iron and Steel Inst., Aug., 1926. Advance proof, 58 pp.).—The equilibria of the reaction $3Fe + 2CO \rightleftharpoons Fe_3C + CO_2$ has been determined between 700° and 1100° by following analytically the variations in carbon content of thin milled cuttings of pure carbon steel which had been heated in a current of carbon dioxide and carbon monoxide of constant composition. Below Ac1 (720°) a bivariant equilibrium was found with ferrite and cementite as solid phases. Above 720° two bivariant equilibria occurred, given by the lines ferrite–austenite and cementite–austenite and between them an infinite number of equilibria for varying content of carbon in the austenite. At 700° the equilibria coincide closely with the experimental equilibria of Schenck (B., 1926, 633), but his extrapolated figures for higher temperatures were not corroborated by direct determination. Decarburisation of steel cuttings in a current of hydrogen was apparent at 600°, increasing rapidly above 650° to a maximum at 950°. The carbon content (0·58%) of a steel maintained at 1050° decreased to 0·35% in 8 hrs. 0·21% in 16 hrs., and 0·13% in 24 hrs. Manganese, nickel, tungsten, and chromium–nickel steels showed about the same tendency to decarburise as pure carbon steels, but chromium steels were more resistant and silicon steels considerably less. In pure nitrogen at 1050° no decarburisation was found other than that associated with gaseous and solid inclusions in the steel. The results of the surface decarburisation of steel between 650° and 1100° in a mixture of carbon dioxide and carbon monoxide, and also in air, are discussed.

C. A. KING.

Treatment of steel with ferro-carbon-titanium. G. F. COMSTOCK (Iron and Steel Inst., Aug. 1, 1926. Advance proof, 9 pp.).—An account of the practical use of ferro-carbon-titanium for deoxidising steel. The alloy as used contains about 17% Ti and 7·5% C, and although of lower deoxidising capacity than 50% ferro-silicon, the stronger affinity of titanium for oxygen enables it to be used as a final addition after silicon or other deoxidisers. In effervescing steels over-oxidation may be corrected, leaving a cleaner steel with less segregation than when aluminium is added. In this connexion the fluxing power of titanium oxide on the slag inclusions in steel is a marked advantage.

C. A. KING.

Initial temperature and mass effects in quenching [of steels]. H. J. FRENCH and O. Z. KLOPSCH (Trans. Amer. Soc. Steel Treat., 1926, 9, 33—74).—The rate of cooling at the centres of spheres, rounds, and plates of high-carbon and alloy steels was determined on quenching from 875° in water, 5% sodium hydroxide solution, oils, and air. The best single measure of the hardening power was found to be the cooling velocity at 720°.

T. H. BURNHAM.

Characteristics of cooling curves [for steel]. H. J. FRENCH and O. Z. KLOPSCH (Tech. Papers, U.S. Bur. Standards, 1926, **20**, [313], 365—385).—From time–temperature cooling curves taken at the centre of variously shaped steel samples, quenched in different media, the following equation is derived: $T - x = y\,(W/S)^n$, where T is the cooling time of the centre, S the surface area of the specimen, W its volume, y a time constant, n a constant depending on the nature of the cooling medium, and x the lag factor (time required for the centre to cool through 2% of the cooling range). The last-named varies with the surface per unit volume.

A. R. POWELL.

Physical investigation into the cause of temper-brittleness [of steels]. J. H. ANDREW and H. A. DICKIE (Iron and Steel Inst., Aug., 1926. Advance proof, 38 pp.).—In steels susceptible to temper-brittleness, a moderate rate of cooling (2° or 3° per min.) from the

tempering temperature causes a considerable fall in specific volume and in hardness, accompanied by comparatively small variations in impact value. The magnitude of the physical change is proportional to the degree of brittleness which may be produced by very slow rates of cooling. In steels of high susceptibility to brittleness, such as nickel–chromium steels, the change in specific volume and hardness is very distinct, but in nickel and chromium steels which are not so susceptible, the change is small. In nickel steel the magnitude of the change increases with the phosphorus content. As an explanation of the phenomenon of temper-brittleness, it is suggested that at higher tempering temperatures, ferrite may dissolve appreciable amounts of carbide (either iron carbide or a double carbide of iron and chromium) which is retained in solid solution by quenching. On cooling slowly from the tempering temperature the carbide is deposited from solution at the grain boundaries to form a brittle network throughout the mass. Certain special elements such as nickel, manganese, chromium, and phosphorus seem to increase the solubility of carbide in ferrite at temperatures near the Ac1 range and to cause it to separate again at lower temperatures on slow cooling, whereas small quantities of molybdenum eliminate brittleness by causing the retention of carbide in solid solution irrespective of the rate of cooling. M. Cook.

Mechanical properties of four heat-treated spring steels. G. A. Hawkins, D. Hanson, and (Miss) G. W. Ford (Iron and Steel Inst., Aug., 1926. Advance proof, 26 pp.).—Results of tests on 0·6% C, 0·8% C, silico-manganese and chrome–vanadium spring steels showed that the quenching temperature of 0·6% C steel was high, but that elongation and reduction of area were lower than for other steels of similar hardness. At a hardness (Brinell) figure of 450 the fatigue limit was as high as ± 47 tons per sq. in. as compared with ± 43·5 for chrome–vanadium, and ± 50 for water-quenched silico-manganese steels. Izod values were lower than for the alloy steels. The 0·8% C steel could not be hardened properly in oil even by quenching from 950°. Tempering at 550° reduced the hardness, tensile strength, and fatigue limit and increased ductility. A more uniform material was produced by quenching from 900° than from 830° though without marked differences in mechanical properties. When tempered above 400° the decrease in hardness of silico-manganese steel was more rapid than for the other steels, and water-quenching gave high values for the limit of proportionality for tempering temperatures of 500°, 550°, and 600°, the results being generally slightly superior to those obtained by oil-quenching. Except when tempered at 600°, chrome–vanadium steel possessed lower values of fatigue limits than silico-manganese steel treated similarly, but the Izod values were superior to those of all the other steels. Chrome–vanadium steel tempered at 600° (Brinell no. 400) combined a high Izod value and good reduction in area, with a fatigue limit of ± 42·5 tons per sq. in. C. A. King.

Effect of phosphorus on the endurance limit of low-carbon steels. F. F. McIntosh (Min. and Met., 1926, 7, 332—333).—Although phosphorus is regarded as an impurity in steel, and is generally considered to exert a deleterious influence on the properties of the metal, a consideration of the results obtained by Stead, Campbell, and others shows that within limits, the strength and fatigue-resisting properties of steel improve as the phosphorus content increases, without causing a proportional loss in ductility. This has been confirmed by tensile, hardness, and fatigue tests on basic open-hearth steel, containing 0·10% C and phosphorus between 0·01 and 0·10%, which show increasing strength, hardness, and ability to withstand fatigue in direct proportion to the phosphorus content. For each increment of 0·01% P the strength increases by 850—1000 lb. per sq. in., and the endurance limit increases 820 lb. per sq. in. Phosphorus up to 0·085% in steels containing 0·09—0·11% C does not manifest itself in any peculiarity of microstructure nor do the carbonising qualities of the steels show any effect traceable to phosphorus. M. Cook.

Testing of hardened steel. A. Lundgren (Iron and Steel Inst., Aug., 1926. Advance proof, 37 pp.).—Methods of testing the mechanical properties of hardened tool steel which are more rapid than practical cutting tests, include a form of bending test, from which the limit of elasticity, the limit of proportionality, and the ultimate strength are derived, the Charpy impact test, and the Rockwell hardness test. Carbon steels containing 1·3, 1·0, 0·9, and 0·6% C have been used in the investigation. The two steels containing 1·3 and 1·0% C were subjected, prior to hardening, to various annealing treatments to obtain various structural forms which have been correlated with mechanical properties, determined after hardening and tempering. The effect of quenching temperature and tempering temperature is considered in detail. With very rapid cooling in benzene after tempering at 300° a much lower bending strength is obtained than by cooling in air after tempering at the same temperature. M. Cook.

Electrochemical potentials of carbon and chromium steels. C. Benedicks and R. Sundberg (Iron and Steel Inst., Aug., 1926. Advance proof, 39 pp.).—The electrochemical potentials of some carbon and chromium steels have been determined in neutral oxygen-free 0·82*N*-ferrous sulphate solution (potential $= E_H$) and in the same solution oxidised with hydrogen peroxide (potential $= E_0$). The value of E_H was found to be higher (more negative) than that of E_0. For pure iron in *N*-ferrous sulphate solution $E_H = -0·708$ volt, and $E_0 = -0·62$ volt. Increasing carbon up to 0·9% lowered the value of E_H, whilst E_0 was increased. For higher carbon contents, the effects were reversed. Quenching lowered the E_H values, but raised the E_0 values up to 0·9% C. The difference between E_H and E_0 tends to vanish for high carbon contents. Differential aeration will therefore only have a slight effect on the corrosion of a hardened high-carbon steel. E_H rises with the addition of chromium up to 8%, and then falls showing a marked minimum at 13—14% Cr, in unquenched specimens, but rises steadily in quenched specimens. The value of E_H in quenched steels containing 13% Cr is much lower with a high carbon content. In unquenched specimens E_0 is unchanged by the addition of chromium, but may be lowered to + 0·31 volt

in quenched specimens. Stainless steel immersed in ferrous sulphate was found to be light-sensitive, and it is suggested that light influences the rusting of iron.

C. J. SMITHELLS.

Anomalies in heat conduction as investigated in spherical steel specimens, with some determinations of thermal (and electrical) conductivity in iron and carbon steels. C. BENEDICKS, H. BÄCKSTRÖM, and P. SEDERHOLM (Iron and Steel Inst., Aug., 1926. Advance proof, 46 pp.).—Measurements of the temperature gradient in different radial directions in hollow spherical specimens of steel showed variations in the ratio of 1 to 8·5 in some cases. The existence of these variations in thermal conductivity was confirmed by thermoscopic observations using a mercury copper iodide indicator, and by direct measurements of thermal distribution. Similar irregularities were found in electrical resistance, though on a much smaller scale, the ratio not exceeding 1 to 1·05. From this it is deduced that the accepted proportionality between thermal and electrical conductivities does not always exist. The irregularities are explained by assuming that thermal conductivity depends on two phenomena, (i) purely thermal, (ii) thermo-electric. The theory previously suggested by Benedicks (Ann. Physik, 1918, [iv], **55**, 1—80) is developed. A method for the determination of the thermal conductivity (λ) is described. A close relation between thermal and electrical resistivities was found for some carbon steels, but not that corresponding with the Wiedemann–Franz law. The thermal resistivity of carbon steel is given by the equation

$$1/\lambda = 4 \cdot 4 + 8 \cdot 7 \Sigma C,$$

where Σ C is the carbon value in weight per cent. of added elements. This gives for pure iron the value $\lambda = 0 \cdot 227$ cal. per cm. sec. deg. The elements are placed in the following order with respect to their effect (increasing) in lowering thermal conductivity in steel: nickel, manganese, hardening carbon, aluminium, silicon. Cementite carbon exerts only a slight influence.

C. J. SMITHELLS.

Chemical composition of tool steels. J. P. GILL and M. A. FROST (Trans. Amer. Soc. Steel Treat., 1926, **9**, 75—98).—The analyses of seventy-five tool steels are critically considered. In carbon tool steel the sulphur and phosphorus should not exceed 0·035%. Non-deformable oil-hardening tool steels are improved by the addition of 0·5% of chromium and tungsten. For finishing cuts steels containing over 1% of carbon and 0·5% of tungsten are recommended. Uniform hardening is assisted by the addition of 0·5% of chromium. For hot-working dies semi-high-speed steels are satisfactory, but should not be hardened in water. Cold-drawing dies are generally made of steel containing 1·5—2·5% C and 11—15% Cr. The effect of additions of molybdenum, uranium, and cobalt to standard high-speed steels (18% W, 4% Cr, 1% V) are discussed and also the rôle of the principal impurities. The authors consider that efforts to standardise tool steel specifications would be detrimental.

T. H. BURNHAM.

Cutting power of high-speed steel tools and methods of testing. F. RAPATZ (Stahl u. Eisen, 1926, **49**, 1109—1116).—The cutting power of tungsten steels increases with the hardening temperature, slowly between 1000° and 1170° and then rapidly to a maximum at about 1300°. The best cutting properties are obtained when the microstructure consists entirely of evenly oriented, fairly large polyhedral grains containing regularly distributed, small inclusions of the ledeburite eutectic, *i.e.*, when the maximum amount of carbide possible is retained in solid solution. With plain 14—18% W steels this structure is obtained by heating for 10 min. at 1250° or for 1—3 min. at 1300°. The longer period of heating at 1250°, although it gives slightly the more satisfactory structure, has the disadvantage that it is difficult to prevent serious oxidation taking place; on the other hand, care must be taken not to exceed 3 min. at 1300° or to allow the temperature to rise more than 20° above this temperature, otherwise the metal will be ruined owing to fusion and redistribution of the eutectic accompanied by abnormal growth of the polyhedral grains. Alloys containing cobalt or vanadium in addition to tungsten may safely be heated to a higher temperature as these elements appear to raise the m.p. of the eutectic. All high-speed tool steels are rendered harder and tougher by subsequent tempering at 580°, whereby the austenite is converted into martensite and internal strains are relieved. As the tensile strength of the alloy increases its life as a cutting tool is reduced very rapidly; an increase in the speed of cutting also reduces the life of the tool but the reduction is not proportional to the cutting speed. The composition of a steel is no indication of its behaviour as a cutting tool; the controlling factors in determining the life of a tool are hardness combined with toughness and suitable microstructure, as well as homogeneity and freedom from slag inclusions.

A. R. POWELL.

Constitution of iron–silicon alloys. G. PHRAGMÉN (Iron and Steel Inst., Aug., 1926. Advance proof, 8 pp.; cf. B., 1925, 285).—*X*-Ray and microscopical examinations show that three intermediary phases occur in the system; two of them which are of variable composition approximately correspond in composition to the compounds FeSi and $FeSi_2$. These two phases give rise to three eutectics, namely FeSi–α-phase, FeSi–$FeSi_2$, and $FeSi_2$–Si, which contain about 21·2, 48·2, and 61% Si respectively. The hypothesis of Oberhoffer that in the presence of sufficient silicon (more than 3·5% is found to be necessary) the α-range in iron disappears and the α- and δ-ranges unite, has been confirmed. The view of Murakami that the third phase corresponds in composition to the formula Fe_3Si_2 and is formed at 1000° by reaction in the solid state is also confirmed.

M. COOK.

Alloys of iron and chromium. E. C. BAIN (Trans. Amer. Soc. Steel Treat., 1926, **9**, 9—32).—Two series of alloys of iron with chromium up to 35% and containing 0·1—0·26% and 0·26—0·50% of carbon respectively were produced from Armco iron and low-carbon ferro-chrome, and subjected to metallographic examination after quenching from seven different temperatures between 900° and 1450°. Quenched taper-heated bars

were also used as corroborative evidence of structural changes effected by temperature. The first group of alloys, containing 3—14% Cr, was martensitic if quenched from above 900°, but at the top end of the range, *e.g.* above 12% Cr, with 0·35% C, δ-iron solid solution was preserved unchanged at room temperature on quenching from 1420°. The α–γ transition point was raised and the γ–δ change lowered with increasing chromium content. In group II with 14—25% Cr γ-iron is not formed on heating except as influenced by the carbon content, the effect of which is marked. High-carbon alloys of the second group were martensitic on quenching from 950—1000°, more austenite being retained and the δ-iron transformation raised to 1350—1400°. Alloys in group III, containing 25—35% Cr, showed no transformation unless the carbon was high, the δ-iron and α-iron solid solutions being continuous. Charts are given showing the temperature of development of the various constituents as affected by the chromium and carbon contents, also the hardness of the high-carbon series in relation to the quenching temperature. Up to 0·5% of carbon was soluble in δ-iron, but on reheating the excess carbon was precipitated within the grains. The austenite in the low-carbon chromium alloys required a high temperature for decomposition but was not resistant to cold work.

T. H. Burnham.

Effect of nitrogen on chromium and some iron–chromium alloys. F. Adcock (Iron and Steel Inst., Aug., 1926. Advance proof, 10 pp.).—Nitrogen was passed over the surface of molten pure iron, chromium, and iron–chromium alloys in a high-frequency induction furnace, and the metals were examined in respect of microstructure and hardness. Nitrogen was absorbed rapidly by chromium up to the extent of 3·9%, but only a small quantity of nitrogen (0·02%) was retained by iron. With alloys of iron and chromium the amount of nitrogen retained increased with the chromium content. In alloys containing about 12% Cr the presence of nitrogen produces a martensitic structure resembling that of iron–carbon alloys, and the hardness may be modified by heat treatment from 115 to 315 Brinell. With higher proportions of chromium (20—60%) alloys containing nitrogen usually present a two-phase structure, one constituent being of sorbitic or pearlitic type, absent in the alloy free from nitrogen, or in a similar alloy containing 0·05% C. Penetration of nitrogen in these alloys was especially rapid in the vicinity of crystal grain boundaries.

C. A. King.

Thermal methods for assaying gold and platinum. J. A. Pinto (Rev. Fac. Cien. Quím., 1926, **4**, 95—140).—A critical review of methods for assaying gold and platinum. A detailed account is given of the methods used by the author, for which the original paper should be consulted.

G. W. Robinson.

Action of hydrogen on hot solid copper. C. S. Smith and C. R. Hayward (Inst. Metals, Sept., 1926. Advance copy, 20 pp.).—The brittle state assumed by copper when it is heated in hydrogen (gassed), and which is considered to be due to steam formed from the hydrogen and the oxygen of the cuprous oxide, has been studied in the case of cast copper. Lengths of copper wire, 4 in. by 0·073 in. diam., containing 0·03 to 0·04% of oxygen, were heated in hydrogen at various temperatures up to 1050° in an electric furnace. The products showed severe embrittlement at 700—800° followed by a marked recovery in strength and ductility at higher temperatures. The recovery is considered to be due to a sintering at higher temperatures which causes the cracks or voids formed during the gassing to close up. To avoid the passing of copper into this brittle state it should not be annealed above 400° in a reducing atmosphere (heating for 30 min. at 550° is sufficient to make the metal quite brittle). The penetration of hydrogen into cast copper was studied on cylinders of the metal, 1·25 in. in diam. and 1·75 in. long. After heating and cooling the expansion was measured and the depth of penetration found from an etched section. The value of $100e/2p$, where e = total expansion and p = the penetration (both in inches), is a measure of the loss of density and is termed the "per cent. linear unsoundness." For copper of low oxygen content (0·03%) the penetration rises rapidly and more or less uniformly with the temperature, whilst the "unsoundness" reaches a maximum about 800° and does not vary above 850°. For metal, however, of high oxygen content (above 0·07%) the penetration is abnormal, rises to a maximum at about 800°, and then rapidly falls off. This maximum is less pronounced as the quantity of oxygen decreases. It is suggested that the sintering has closed up the voids and that further penetration can only take place by ordinary diffusion. That it only takes place with high oxygen content is due to the larger amounts of steam evolved, which strain the metal and induce recrystallisation which always precedes sintering. Photomicrographs are given which support this theory. Copper which has been forged after casting does not show abnormal penetration values. The time and penetration curve for low-oxygen copper is almost a straight line, that for high-oxygen copper approaches a parabola. Gassed copper, which is usually considered incapable of restoration unless re-melted, can be converted into metal of good mechanical and electrical properties by rolling to about half the thickness of the gassed material. The ductility is far in excess of that of the original copper, whilst the electrical conductivity of wire of 0·08 in. (after deoxidation and annealing) was 101·1% on the Matthiessen scale. This treatment could be applied to copper bars before rolling into rods.

A. Coulthard.

Season-cracking in arsenical copper tubes. A. Pinkerton and W. H. Tait (Inst. Metals, Sept., 1926. Advance copy, 6 pp.).—Hollow sunk arsenical copper tubes are liable to season-cracking when reductions of sectional area exceed 21%, but sufficient data have not been obtained to fix definitely a safe working limit. Annealing at 240° renders the tubes immune from cracking in mercurous nitrate without affecting the hardness. Arsenic-free copper tubes having average internal stresses of the same order as arsenical copper tubes are not liable to season-cracking.

M. Cook.

Direct production of brass from mixed ores. A. W. Guertler (Metall. u. Erz, 1926, **23**, 325; Chem. Zentr., 1926, II., 824).—By melting sulphide ores of

lead and zinc with metallic copper in excess, copper matte and a mixture of metallic lead, copper, and zinc are formed, which separate, in the liquid state, into a layer rich in lead with little zinc, lead, and a copper–zinc layer with little lead. The last-named is melted with copper and yields a serviceable brass.

C. T. GIMINGHAM.

Working of brass at high temperatures. K. HANSER (Z. Metallk., 1926, 18, 247—255).—Diagrams have been constructed on the equilibrium diagram of copper–zinc alloys containing more than 50% Cu showing lines of equal compressibility (under static and dynamic pressure), hardness, tensile strength, elongation, and reduction of area. The reduction in height of β-brass under a dyamic blow remains fairly constant up to 500°, then increases rapidly with rise of temperature, whereas with α-brass a slow but steady increase takes place throughout the temperature range. Under the conditions of the ordinary compressibility test the maximum brittleness is shown by alloys containing 8—20% Zn between 200° and 500°, but no sign of fracture occurs until the height has been reduced by 50—55%. The hardness curves are similar to those of compressibility, but the impact test figures are lowest between 300° and 500° for all alloys with more than 3% Zn. The tensile strength of all the brasses falls fairly regularly with rising temperature, but the reduction in area of α-brass decreases to a minimum between 300° and 500° according to the composition, then increases to its original value above 700°, whereas the greatest reduction in area with β-brass occurs just above 500° and the minimum at the ordinary temperature. These results show that the reduction in area is the best indication of the most suitable temperature for working brass. Further tests on brass containing 67% and 60% Cu show that, when rolled above 500°, the metal is stronger the more rapid the passage through the rolls.

A. R. POWELL.

Copper–magnesium alloys. W. T. COOK and W. R. D. JONES (Inst. Metals, Sept., 1926. Advance copy, 14 pp.).—Alloys of magnesium and copper containing from a trace to 10% Cu have been prepared by melting the metals in bottom-pouring forged-steel crucibles with a flux of magnesium chloride and fluoride (3—4% of the weight of the metal) and casting into $\frac{3}{4}$ to $1\frac{1}{8}$ in. (diam.) moulds, under conditions readily reproducible in workshop practice. Gas-holes of $\frac{1}{16}$—$\frac{1}{8}$ in. in diam. which appeared on the first casting were eliminated by allowing the metal to cool slowly below the solidification temperature, then immediately re-melting and re-casting (cf. Archbutt, B., 1925, 286). The melting losses did not exceed 0·75%. Tables and graphs are given showing the tensile strengths of the chill-castings as determined on a Riehlé multiple-lever testing machine. The alloys have a comparatively low tensile strength, little ductility, and a low limit of proportionality. The addition of copper up to 2 or 3% to magnesium is beneficial. Brinell hardness tests carried out on an Amsler machine show the hardness to increase with the amount of added copper. The hardness does not appear to increase on ageing. The alloys have a low impact value. Etching with an alcoholic solution of 1% nitric and 0·5% hydrochloric acids showed the alloys to consist of grains of almost pure magnesium in a eutectic of the composition, Mg_2Cu, which had a tendency to become globular. The macrostructure developed by a 5% alcoholic solution of nitric acid shows a refinement of the grain due to copper.

A. COULTHARD.

Influence of the thermal and mechanical treatment of aluminium on its resistance to corrosion. W. WIEDERHOLT (Korrosion u. Metallschutz, 1926, 2, 126—133; Chem. Zentr., 1926, II., 938—939).—The velocity of dissolution of aluminium sheet in acid increases with rise of annealing temperature to a maximum at 300°, at which temperature the maximum separation of impurities from solid solution in the aluminium occurs, with the consequent formation of the greatest number of local elements. Above 300° the impurities, especially silicon, diffuse into the metal to form a solid solution, and the corrosion rate decreases proportionately. Below 300° the impurities are thrown out of the solid solution at a rate proportional to the time of annealing, hence the rate of corrosion of metal annealed below 300° is also proportional to the time of annealing. The resistance to corrosion is increased by annealing in nitrogen, and to a greater extent by annealing in oxygen. The coarser the crystalline structure after annealing, the higher is the resistance to corrosion, so that the degree of deformation to which the metal has been subjected by cold-work has a considerable influence on the resistance of the subsequently annealed metal to corrosion. Reductions of 2—15% have practically the same effect in this respect, but with a greater reduction the corrodibility increases with the degree of reduction.

A. R. POWELL.

Constitution and structure of commercial aluminium–silicon alloys. A. G. C. GWYER and H. W. L. PHILLIPS. **Properties of modified aluminium–silicon alloys.** D. STOCKDALE and I. WILKINSON (Inst. Metals, Sept., 1926. Advance copy, 43 pp.).—The composition of the normal silicon–aluminium eutectic is confirmed to be 11·7% Si and 88·3% Al, and its m.p. is 577°. After addition of a modifying agent the all-eutectic alloy contains 12·85% Si and solidifies at a lower temperature, varying according to the modifying treatment and the rate of cooling, but it always melts at 577°. The higher the proportion of modifying agent added the lower is the f.p. of the alloy, and the more rapid the cooling the finer is the crystal structure. These facts are explained by assuming that the sodium obtained from the modifying agent acts as a colloid and hinders the separation of crystals from the melt, thereby inducing supercooling. The supercooled alloy then solidifies rapidly in the presence of a colloid, which acts as a protective colloid and retains the aluminium and silicon in the colloidal form in which they first separate from the melt. In substantiation of this theory, it is shown that addition of sodium to aluminium–copper and manganese-aluminium alloys and of aluminium to lead–antimony and antimony–copper alloys also results in a refinement of the structure. The converse of this effect is produced by adding sodium hydroxide to iron–aluminium alloys at 900°, acceleration of the separation

of $FeAl_3$ being induced, followed by the coagulation of this constituent into large crystals. In a similar manner, addition of sodium chloride to the remelted modified silicon–aluminium alloys causes rapid reversion to the normal form, probably owing to its effect in coagulating the colloid. Pronounced colonisation occurs along the grain boundaries of silicon–aluminium alloys modified with excess of sodium hydroxide; the same effect is produced by addition of copper or zinc to the alloys. The silicon–aluminium–iron system contains a ternary eutectic, f.p. 577°, containing 11·6% Si and 0·8% Fe, the iron occurring as the unknown X constituent. Alloys containing more than 11·6% Si also show the presence of a new iron constituent, δ, which appears to be a ternary substance and is converted into X on annealing. D. STOCKDALE and I. WILKINSON, in an appendix (12 pp.), give the results of various mechanical tests on modified silicon–aluminium alloys. Their results in general confirm those of Grogan (*infra*). The alloy containing 11% Si and the minimum amount of iron seems to have the best all-round combination of properties and the smallest tendency to reversion.

A. R. POWELL.

Mechanical properties of silicon–aluminium alloys. J. D. GROGAN (Inst. Metals, Sept., 1926. Advance copy, 13 pp.).—Silicon-aluminium alloys modified by the salts method usually have better mechanical properties than those modified by addition of metallic sodium, owing to their greater soundness. The amount of sodium retained by the modified alloys varies from 0·002 to 0·008%. The density of the alloys decreases linearly with the silicon content from 2·68 for the 8% Si alloy to 2·65 for the 14% alloy; the stress required to produce a permanent deformation of 0·5% increases regularly from 5—6 tons per sq. in. for the 8% alloy to 7·8 tons for the 14% alloy chill-cast, and from 4·8 tons to 6·5 tons respectively for the corresponding sand-cast alloys, and the ultimate stress increases from 12·1 tons per sq. in. for the 8% chill-cast alloy to 13·4 tons for the 12% alloy, a further 2% Si making little difference. The corresponding ultimate stress figures for the sand-cast alloys are 10·3 and 11·6 tons per sq. in. respectively. The elongation falls rapidly from 19% for the 8% Si alloy chill-cast and from 16·5% for the same alloy sand-cast to 11 and 9·5% respectively for the 12% alloy, further silicon up to 14% making no difference. The Charpy impact values fall with increase of silicon from 8 to 13·5% from 1·34 ft.-lb. to 0·56 ft.-lb. for chill-cast and from 0·80 ft.-lb. to 0·44 ft.-lb. for sand-cast alloys. The fatigue range of the alloy containing 8·5% Si and 0·3% Fe is $\pm$ 4·2 tons per sq. in. The hardness increases linearly with the silicon content from 8 to 14% from 50 to 65 for chill-cast and from 44 to 57 for sand-cast alloys. Addition of zinc increases the hardness and tensile strength, but reduces considerably the ductility, whereas magnesium does not affect the tensile strength but ruins the ductility.

A. R. POWELL.

Silumin and its structure. B. ÔTANI (Inst. Metals, Sept., 1926. Advance copy, 25 pp.).—The solubility of silicon in solid aluminium, as determined by chemical methods and by electrical conductivity measurements, is 1·47% at 550° and 0·43% at 360°. The most satisfactory modified structure using metallic sodium results from the addition of 0·1% Na at 720—750°; using "salts," the best results are obtained with 1% of a mixture of 40% of sodium fluoride and 60% of potassium fluoride at 750°, and by 0·5—0·8% of sodium hydroxide at 700—800°. A structure resembling the modified structure may be obtained by quenching the alloy immediately after solidification, so that the exterior layers of chill-cast normal alloys often exhibit a fine-grained eutectiferous structure. The action of sodium in modifying the structure of silicon–aluminium alloys is discussed at some length and evidence is adduced to show that none of the existing theories fits in with all the known facts. An explanation is suggested, based on the fact that a second liquid consisting almost wholly of metallic sodium exists in the melt during its passage through the solidification range; this liquid separates out from its solution in the aluminium just as the aluminium and silicon crystals begin to form, and thereby hinders their growth and causes the formation of many more nuclei. Bismuth and cadmium bring about only a slight modifying effect, although they also form no solid solutions with aluminium; they are, however, less soluble than sodium in the molten alloy, and have relatively high densities. Hence their restraining influence on crystallisation is slight.

A. R. POWELL.

Significance of silicon in the ageing of aluminium alloys containing lithium or magnesium. P. ASSMANN (Z. Metallk., 1926, 18, 256—260).—The maximum hardness of alloys of lithium or magnesium with commercial aluminium, after quenching from above 500° and ageing at 18°, is obtained when the silicon impurity of the aluminium is just sufficient to form the silicides Li_3Si or Mg_2Si; with more or less silicon than this the hardness of the aged alloy decreases proportionately to the excess or deficiency. With very pure aluminium neither lithium or magnesium produces ageing phenomena, thus indicating that the ageing properties of these alloys are due to the solubility of the silicide at 18° being less than that at 500°. Whilst the hardening effect of magnesium silicide is not affected by ageing at 100°, but the rate of hardening is increased, a similar treatment of alloys containing lithium silicide results in a softer alloy than is obtained by ageing at 18°. Annealing at 200° in each case destroys the effect of ageing. Copper up to 4% and zinc up to 12% increase the hardness of the lithium alloys aged at 18°, but lithium reduces the hardness of copper–aluminium or zinc–aluminium alloys aged at 100°. The maximum hardness at 18° of the lithium alloys is obtained with 1·6% Li_3Si, and of the 4% Cu or 12% Zn alloys with 0·67% Li. Thus it appears that copper and zinc depress the solubility of Li_3Si in aluminium at 500°, and that Li_3Si tends to repress the "artificial" ageing effect of copper and zinc.

A. R. POWELL.

Constitution and physical properties of alloys of cadmium and zinc. C. H. U. JENKINS (Inst. Metals, Sept., 1926. Advance copy, 35 pp.).—The physical properties of alloys of zinc and cadmium have been found to be more complex than would result from a simple eutectiferous series of these metals, and the

complexity has been traced to alterations in the alloys caused by the two polymorphic changes (especially the one about 350°) which appear to occur with zinc. The eutectic point is at 266° with Cd 82·6%. Although a considerable degree of metastability occurs in all the alloys no intermediate or solid solutions are formed, and no marked thermal arrests were observed except at the temperature of the liquidus and the eutectic point. The physical properties of the cadmium-rich, zinc-rich, and eutectic alloys have been examined, and tables of values are given for the tensile strength, Brinell hardness, and electrical conductivity of cast, annealed, and aged alloys. Somewhat in opposition to the views of Rigg and Morse (B., 1915, 1097) the properties of cast and rolled zinc are found to be improved by the addition of small amounts of cadmium, although some degree of hot-shortness occurs in alloys containing more than 3%. In the zinc-rich alloys a non-permanent hardening effect was observed which is followed by softening at room temperature over a period of months. The cadmium-rich alloys soften to minimum values much more rapidly. Cold-worked cadmium has been found to recrystallise completely at the temperature produced by cold working. The solid solubility of cadmium in zinc amounts to 1·75—2% at 250°, that of zinc in cadmium 2—2·25%, but this falls to less than 0·25% of cadmium and 0·75—1·0% of zinc at 60°. The presence of cadmium in zinc does not appear to accelerate corrosion due to rain-water, but rather the reverse. Zinc containing a small quantity of cadmium is superior for certain purposes, such as zinc-base die-casting and zinc sheeting. The eutectic alloy has a strength of about three times that of ordinary tin–lead solder, and possesses properties which should lead to its increased use as a medium hard solder. A. Coulthard.

Bearing metals. R. T. Rolfe (J. Inst. Metals, 1926, 35, 439—440).—Addition of copper to tin–antimony alloys tends to prevent segregation; thus a 90% Sn, 10% Sb alloy segregates into two layers, whereas no segregation at all takes place in an alloy of 85% Sn, 8·5% Sb, and 6·5% Cu. The tensile strength of an Admiralty tin-base bearing metal increases with the casting temperature, and decreases with an increase in the temperature of the mould. Casting temperature, however, has no effect on the compressive load required to produce a permanent set, but a mould temperature of 200° gives better results than a higher or lower temperature; in practice it appears to be best to cast at 365° into moulds preheated to 200°. Micrographic examination of some specimens of this alloy showed a cuboid of SnSb split as though by a wedge by a needle of the hard $SnCu_3$ compound. A. R. Powell.

Comparison of static and dynamic tensile and notched-bar tests. K. Honda (Inst. Metals, Sept., 1926. Advance copy, 11 pp.).—With tension tests the energy absorbed on impact is generally greater than that absorbed in the statical test. For example, in a series of steels containing 0·1—0·9% of carbon, the work of rupture was determined both dynamically and statically, and showed the following mean ratio: dyn./stat. = 1·37. In the case of bending tests it is confirmed that the same amount of energy is required whether the test is dynamic or static. Deformation is considered the most important factor affecting the difference in the static and dynamic energies absorbed in the tension tests. A method is described by which the degree of fatigue at any given period during an impact test may be determined. A. Coulthard.

Steel age—1876 to 1926. J. A. Mathews (Ind. Eng. Chem., 1926, 18, 913—914).

Fifty years' progress in aluminium. J. D. Edwards (Ind. Eng. Chem., 1926, 18, 922—924).

Metallurgy fifty years ago and now. W. M. Corse (Ind. Eng. Chem., 1926, 18, 892—895).

Effect of zinc, zinc oxide, and zinc sulphide upon the health of workmen. Batchelor and others.—See XXIII.

Patents.

Refining of iron. G. S. Evans (U.S.P. 1,590,731, 29.6.26. Appl., 9.5.25).—The addition of sodium carbonate which has been aggregated to a dense product by fusion and subsequent cooling to the charge of iron, coke, and limestone used in foundry cupolas, produces a strongly alkaline fluid slag which absorbs the greater portion of the sulphur present and forms a good coating on the molten iron. About 1—4 lb. of sodium carbonate are used per ton of metal treated. T. S. Wheeler.

Coating iron or steel articles [with tin–aluminium alloy]. I. J. Koehline (U.S.P. 1,591,983, 13.7.26. Appl., 20.6.24).—Iron or steel articles are coated with an alloy of tin containing up to 20% of aluminium by dipping in a bath of the molten alloy. The alloy is harder, lighter, and cheaper than pure tin and protects the iron better against corrosion. T. S. Wheeler.

Alloy steel. R. T. Wirth (U.S.P. 1,593,924, 27.7.26. Appl., 19.10.21).—An alloy steel contains from 2 to 30% of chromium, and not less than 10% of tungsten or molybdenum or both, the total of alloying elements exceeding 35%. M. Cook.

Treating ingot iron. W. Tafel (U.S.P. 1,594,133, 27.7.26. Appl., 22.8.24. Conv., 16.6.23).—To produce iron or steel similar to wrought iron, a mixture or a slag containing ferrous oxide, ferric oxide, and silica is added to the molten iron in a furnace. C. A. King.

Metallurgical furnace. F. G. Breyer, Assr. to New Jersey Zinc Co. (U.S.P. 1,594,000, 27.7.26. Appl., 4.9.24).—The furnace comprises a heating chamber with a flue for conducting the metal vapour produced in the furnace to the atmosphere outside the furnace, and means for directing a blast of relatively cool gas against the stream of vapour issuing from the flue. A. R. Powell.

Corrosion-resisting metal. C. E. Jones, Assr. to Gen. Electric Co. (U.S.P. 1,594,061, 27.7.26. Appl., 19.6.22).—An article consisting largely of aluminium is electroplated with cadmium, to render it resistant to corrosion in a salt atmosphere. B. M. Venables.

Production of magnesium. H. E. Bakken, Assr. to Amer. Magnesium Corp. (U.S.P. 1,594,344, 3.8.26.

Appl., 1.11.22. Renewed 17.4.26).—Material containing metallic magnesium is heated under a pressure lower than the vapour pressure of magnesium at its m.p. to a temperature at which the metal sublimes readily.

A. R. POWELL.

Production of magnesium. H. E. BAKKEN and W. G. HARVEY, Assrs. to AMER. MAGNESIUM CORP. (U.S.P. 1,594,345, 3.8.26. Appl., 8.5.23).—Pure magnesium is obtained by subjecting material containing metallic magnesium to distillation under a pressure slightly above the vapour pressure of the metal at its m.p. and condensing the vapours evolved.

A. R. POWELL.

Magnesium product. Working magnesium. Sublimation apparatus [for magnesium]. H. E. BAKKEN, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,594,346—8. 3.8.26. Appl., [A], and [B], 1.11.22, [C], 10.2.25).—(A) A crystalline mass of metallic magnesium is obtained by sublimation. (B) This mass may be worked into a coherent product by subjecting it to pressure at a temperature just below its m.p. (C) Impure magnesium is refined by sublimation in a vertical cylindrical retort, the lower portion of which is heated in an electric resistance furnace while the upper part is cooled to allow the metal to condense on the sides. The pressure inside the retort is maintained just below the vapour pressure of the metal at its m.p.

A. R. POWELL.

Electrothermal reduction of alumina. F. C. FRARY, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,594,362, 3.8.26. Appl., 10.4.24).—In the process of producing aluminium–copper alloys by the electrothermal reduction of alumina over a bath of molten copper, the contents of the furnace are tested periodically to determine whether aluminium carbide is present. If such is the case alumina alone is added to the furnace charge so as to react with the excess of carbide.

A. R. POWELL.

Refining metals. J. L. MULLIGAN, Assr. to UNITED STATES SMELTING, REFINING AND MINING CO. (U.S.P. 1,594,424, 3.8.26. Appl., 19.6.24).—In the method of refining a molten metal by treating it with a fused alkali hydroxide, a thickening agent is added to the slag just previous to skimming. A. R. POWELL.

Concentration of ores. J. L. STEVENS (U.S.P. 1,594,858, 3.8.26. Appl., 23.8.24; cf. U.S.P. 1,525,211, B., 1925, 300).—The reaction product of fusel oil, carbon disulphide, and alkali is added to the mineral pulp and the mixture subjected to flotation.

S. BINNING.

Production of single-crystal metal wires from liquid metal. W. FREY and E. VON GOMPERZ (G.P. 429,276, 23.11.21).—The bath of liquid metal is maintained at a temperature just above the m.p., and is cooled evenly at one spot to the m.p. A wire of the same metal is dipped into the liquid at this spot and slowly withdrawn, whereby a single-crystal wire is obtained.

A. R. POWELL.

Melting and refining of readily oxidisable metals (e.g., copper, lead, tin, zinc, or aluminium) and their alloys. P. T. ARNEMANN (G.P. 429,666, 27.2.25).—Readily oxidisable metals are melted in a crucible by means of neutral or reducing gas flames directed on to the surface of the metal at such a pressure that they completely envelop the crucible and prevent access of air. For refining purposes slightly oxidising flames are used in the same way. High melting temperatures may be obtained by this method without any loss of valuable metal by oxidation. A. R. POWELL.

Increasing the amount and phosphoric acid content of basic slag. H. HILBERT (G.P. 429,767, 23.9.25. Addn. to 418,102; cf. B., 1926, 162).—A mixture of calcium phosphate and calcium oxide is used in place of calcium phosphate alone to replace the limestone or dolomite added in the converter process. The formation of iron phosphate is prevented.

C. T. GIMINGHAM.

Treatment of complex ores and smelter products. F. KRUPP GRUSONWERK A.-G. (G.P. 429,938, 6.4.24).—Complex ores are subjected to a blast of air in a combustion chamber in such a way that the oxygen supplied to the zone in which decomposition of the sulphides takes place is sufficient only for the combustion of the sulphur, so that the easily volatilised metals are not oxidised, and are eliminated from the charge while it is still suspended in the blast. A. R. POWELL.

Lead alloy, especially for cable covering. A. H. R-SUNDIUS (Nor. P. 40,552, 19.7.23).—A lead alloy contains tin, bismuth, and antimony or arsenic in addition to magnesium. The total amount of metal alloyed with the lead may be as high as 2%, but, preferably, should not be more than 0·5%. C. T. GIMINGHAM.

Purification of iron. MATHIESON ALKALI WORKS, Assees. of G. S. EVANS (E.P. 250,522, 6.7.25. Conv., 10.4.25).—See U.S.P. 1,590,739; B., 1926, 753.

Complete elimination of zinc from fine zinciferous ores and the like, especially zinciferous purple ores or calcined residues. A. L. MOND. From METALLBANK & METALLURGISCHE GES. (E.P. 257,133, 17.11.25).—See G.P. 421,384; B., 1926, 412.

Coating articles particularly with metals [by the spray process]. M. U. SCHOOP (E.P. 236,175, 18.5.25. Conv., 30.6.24).

Briquette (U.S.P. 1,590,706).—See II.

Alloy for glass-melting container (U.S.P. 1,592,336).—See VIII.

Electric furnaces for bright annealing (E.P. 242,283).—See XI.

XI.—ELECTROTECHNICS.

Inside frosting of incandescence [electric] lamps. M. PIPKIN (Ind. Eng. Chem., 1926, **18**, 774—776; cf. E.P. 228,907, B., 1925, 804).—Frosting of the inner surface of lamp bulbs was obtained by repeated internal spraying at 5-sec. intervals to a total of 60 secs. with a mixture of water, barium sulphate, sodium bisulphate, ammonium bifluoride, dextrin, and hydrofluoric acid.

(11·9% by weight) at 50°, followed by a strengthening treatment consisting of a similar spraying with a mixture of water, barium sulphate, dextrin, and hydrofluoric acid (7·44% by weight). A suitable commercial frosting mixture was made from 48—60% hydrofluoric acid saturated with ammonium bifluoride with the addition of enough water and sodium carbonate to reduce the acidity to 25%, the bulb being covered and left for 25—50 secs. at 30—50°. Fine-grained etching was given by the mixture: ammonium bifluoride 42%, dextrin 7%, barium sulphate 20%, sodium bisulphate 3·5%, hydrofluoric acid 27·5%, water to give an acidity of 18—25% of hydrofluoric acid. Internally frosted bulbs had about the same diffusion as those externally frosted, but they absorbed less light. The absorption of light, in the case of daylight lamps, was greatly increased by internal frosting.

A. COUSEN.

Theoretical study of the yield of the high-frequency electric furnace with alternating current. G. RIBAUD (J. Phys. Radium, 1926, [vi], 7, 250—256).

A half-century of artificial [electric] lighting. M. LUCKIESH (Ind. Eng. Chem., 1926, 18, 920—922).

Electrical resistance of refractory materials. FERGUSON.—See VIII.

Electro-chemical potentials of carbon and chromium steels. BENEDICKS and SUNDBERG.—See X.

Thermal and electrical conductivity in iron and carbon steels. BENEDICKS, BÄCKSTRÖM, and SEDERHOLM.—See X.

Determination of electrical conductivity of milk. GERBER.—See XIX.

PATENTS.

Gaseous electric conduction devices. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of E. E. CHARLTON (E.P. 237,235, 2.7.25. Conv., 17.7.24).—To improve the efficiency of electric discharge devices which operate by the ionisation of a gas, the voltage necessary to produce discharge between the electrodes may be reduced to a minimum by providing in the bulb, together with an inert gas, an oxide of an alkali metal preferably associated with a highly oxidisable metal such as an alkaline-earth metal. Methods of introducing the alkali and alkaline-earth metals are described. (Reference is directed, in pursuance of Sect. 7 (4) of the Patents and Designs Acts, 1907 and 1919, to E.P. 224,544).

M. E. NOTTAGE.

Oxide cathodes for discharge tubes. N. V. PHILIPS' GLOEILAMPENFABRIEKEN (E.P. 245,147, 23.12.25. Conv., 27.12.24).—A wire or other body of tungsten or a similar metal or alloy is at least partly oxidised at its surface and is surrounded by the vapour of an alkaline-earth metal to deposit alkaline-earth oxide on the surface. In one method of carrying out the process an easily decomposable alkaline-earth compound, such as barium azide, is introduced into the tube, and during the process of exhausting the tube is heated sufficiently to effect its decomposition and volatilise the metal. H. HOLMES.

Electric discharge devices. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of D. A. MULLANEY (E.P. 250,906, 13.1.26. Conv., 16.4.25).—In a device consisting of an evacuated envelope containing co-operating electrodes, the thoriated cathode of which is very sensitive to minute quantities of gas, a quantity of tantalum is arranged so as to be heated during operation. To this end it may constitute part of one of the electrodes, being preferably fixed to the central part of the anode or, in the case of a device containing a grid or controlling electrode, this may consist of tantalum. The tantalum exercises a cleaning-up action for gas during the entire life of the device. M. E. NOTTAGE.

Electron-emission material. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,591,717, 6.7.26. Appl., 28.9.22).—A refractory alkaline-earth oxide, such as barium oxide, is mixed with a metallic powder, *e.g.*, tantalum or platinum, and coated on a suitable wire, *e.g.*, platinum-iridium, by suspending the coating mixture in nitrocellulose dissolved in amyl acetate and removing the organic material by the action of heat from the coated wire. Alternatively, mixtures of tantalum and the oxide may be converted into a filament by any of the methods usually employed for the pure metal. The electron-emission material obtained retains its activity for a long period. T. S. WHEELER.

Preparation of rare metallic oxides [for electric lamps]. J. A. HEANY (U.S.P. 1,592,459, 13.7.26. Appl., 14.11.18).—Rare metal oxides, such as thoria or zirconia, when associated with the heating element of an electric-light bulb, liberate occluded oxygen which attacks the heating element. To prevent this, the purified oxide is moulded and then immersed in a bath of molten paraffin until the hydrocarbon has replaced the occluded gases in the pores of the material. It is then removed and placed in position with the heating element in the bulb, which is attached to the pump and exhausted with the electric current running. When the pump has removed all the hydrocarbon, the bulb is sealed off or filled as desired.

E. S. KREIS.

Electric incandescent device [lamp]. G. R. FONDA, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,594,057, 27.7.26. Appl., 30.8.24).—To prevent the volatilisation and crystal faulting of the tungsten filament in a gas-filled lamp, zirconium chloride is introduced, so that the filament operates in contact with zirconium chloride vapour at a pressure equal to its vapour pressure at the temperature of the lamp bulb. B. M. VENABLES.

Electric furnaces for bright annealing. SIEMENS-SCHUCKERTWERKE, Assees. of HERAEUS-VACUUMSCHMELZE A.-G., and W. ROHN (E.P. 242,283, 28.10.25. Conv., 1.11.24).—The heating chamber of the furnace consists of a metal container enclosed in an air-tight furnace casing, the electric heater being contained in the space between them and the whole surrounded by a further heat-insulating layer. The heating current is controlled by the thermal expansion of the metal container. The protective gas passes separately or consecutively through the space between the metal container and the furnace casing and through the interior of the

metal container, and also through antechambers which are provided at one or both ends of the heating chamber.
M. E. Nottage.

Electric furnace. J. J. Naugle (U.S.P. 1,593,879, 27.7.26. Appl., 21.4.23).—An electric furnace for treatment of carbonaceous material or the like consists of a container, along the inner periphery of which extends longitudinally a number of electrodes, means being provided for keeping the latter in motion so that they are intermittently immersed in the carbonaceous material.
M. E. Nottage.

Regenerative cell of manganese dioxide, coal, and zinc [feeble current accumulator].—E. Heese (E.P. 254,549, 21.8.25).—A regenerative cell comprises a positive element of pure zinc (as distinct from commercial zinc) and a depolarising mixture enclosed in an osmotic envelope, *e.g.*, of animal skin, fish bladder, etc., and a manganese dioxide–coal negative element. The electrolyte consists of zinc ammonium chlorate or zinc ammonium chloride, and on recharging the cell a surface of pure zinc is formed. J. S. G. Thomas.

Insulated electrical conductors and the like. E. Taylor, Assr. to Taylor Laboratories, Inc. (U.S.P. 1,590,599 and 1,590,608, 29.6.26. Appl., [a] 17.6.24, and [b] 18.3.26).—(a) The conductor is coated with cellulose by applying a coating of cellulose dissolved in zinc chloride or cuprammonia and removing the solvent constituents electrolytically. The apparatus consists of a tank, containing the cellulose solution, fitted with pipes for supplying more solution and maintaining a gas pressure. Through the side wall of this tank is fitted a nozzle and opposite to it, in the other wall, is a nozzle with a bore the size of the object to be coated. This projects into a second tank, containing dilute sulphuric acid, and having a tubular copper cathode. At the end of the cathode is a stuffing-box with a central perforation connecting it to a washing tank. The wire to be insulated passes through the two nozzles in between which it becomes coated with cellulose solution. In the second tank, while passing through the tubular cathode, it acts as the anode, thereby becoming coated with a film of cellulose. (b) The coating of cellulose on the conductor is transparent, homogeneous, and impermeable to oil. R. B. Clarke.

Electrolytic cell [for deposition of cellulose]. E. Taylor, Assr. to Taylor Laboratories, Inc. (U.S.P. 1,590,604, 29.6.26. Appl., 21.6.24).—A rotating hollow drum is mounted so that its porous periphery passes just above the copper base of a semi-cylindrical vessel. Between the copper base and the drum is a solution of cellulose in cuprammonia and inside the drum is a conducting solution. A small curved anode, of platinum if the conducting solution is dilute sulphuric acid, or of carbon if it is ammonium nitrate, is suspended from the centre of the drum; the copper base acts as the cathode of the cell. On passing a current cellulose is deposited on the outer surface of the drum, from which it is scraped and wound on to reels. R. B. Clarke.

Electric-battery construction. E. Taylor, Assr. to Taylor Laboratories, Inc. (U.S.P. 1,590,605, 29.6.26. Appl., 21.6.24).—The electrodes consist of antimony–lead embedded in non-fibrous cellulose mixed with a metal oxide, *e.g.*, manganese dioxide, the whole being covered with a thin layer of permeable, colloidal cellulose. The cell is filled with dilute sulphuric acid and is closed by a cellulose lid. The electrodes are prepared as follows: a solution of cellulose in cuprammonia is evaporated to a thick paste and the manganese dioxide is stirred in. The mass is placed in an electrolytic cell, the anode of which is a grid of antimony–lead. On passing a current cellulose containing admixed manganese dioxide is deposited on the grid which then becomes the anode of a cell containing a pure solution of cellulose in cuprammonia. R. B. Clarke.

Electrolytic apparatus [for deposition of cellulose]. E. Taylor and E. F. Chandler, Assrs. to Taylor Laboratories, Inc. (U.S.P. 1,590,609, 29.6.26. Appl., 19.3.26; cf. U.S.P. 1,590,593, p. 818).—The electrolytic cell is closed to the atmosphere and contains four copper cathode plates. A belt carrying a solution of cellulose in cuprammonia passes over pulleys from the top to the bottom of the cell, underneath the first plate and over the top of the second, and so on.
R. B. Clarke.

Electrolytic apparatus. W. G. Allan, Assr. to F. G. Clarke (U.S.P. 1,592,512, 13.7.26. Appl., 8.4.21. Renewed 7.12.25).—A number of cells are connected to form a battery and divided into working groups. The anode and cathode liquids are removed to separating chambers, where the oxygen and hydrogen are removed. The liquids then flow to a mixing chamber before being returned to the cells. E. S. Kreis.

Apparatus for electric formation of oxides of nitrogen. J. S. Island (E.P. 257,083, 30.7.25).—See F.P. 601,740; B., 1926, 592.

Dry cell. M. Markiewicz and W. Römer (U.S.P. 1,597,165, 24.8.26. Appl., 31.5.24. Conv., 5.7.23).—See E.P. 234,701; B., 1925, 639.

Electric welding process and apparatus. G. B. Ellis. From T. E. Murray (E.P. 257,055, 30.6.25).

See also pages 818, **Conversing fibrous cellulosic material** (U.S.P. 1,590,592—3); **Treating cellulosic material** (U.S.P. 1,590,594); **Producing colloidal cellulose** (U.S.P. 1,590,596). 819, **Treating silk and cellulose material** (U.S.P. 1,590,600); **Treating cellulose** (U.S.P. 1,590,601 and 1,590,606). 833, **Electrothermal reduction of alumina** (U.S.P. 1,594,362). 840, **Production of artificial leather** (U.S.P. 1,590,602).

XII.—FATS; OILS; WAXES.

Determination of fat in oilseed [linseed] and oilcake by the refractometer. H. Zander (Z. Unters. Lebensm., 1926, **51**, 324—335).—25 g. of the sample are ground so that 80% will go through a No. 34 sieve, and 2 g. of the powder are weighed into a mortar, previously warmed to 70°, and triturated for 2 minutes with 4 c.c. of monochloronaphthalene. After filtration a drop is examined in a refractometer, the temperature of each reading being taken. The oil content is obtained from the readings by reference to tables given, which

have been obtained by the examination of pure linseed oil at a temperature of 25°. The refractometer readings for oilseed or oilcake must be brought to this temperature by tables or curves; every degree above 25° necessitates an addition of 0·00045 to the refractometer reading, and every degree below, a corresponding deduction. Results compared with the ether extraction method agreed closely. Methods using trichloroethylene and also glacial acetic acid in an immersion refractometer proved to be unsatisfactory, and the sources of error are discussed.

W. G. CAREY.

Determination of milk fat and coconut oil in fat mixtures and examination of milk sweets. H. FINCKE (Z. Unters. Lebensm., 1926, **51**, 357—368).—The author finds that by employing a modified method for the Kirschner value, and determining the Polenske value in the usual way, it is possible to determine approximately the coconut oil content. 5 g. of the fat, with the addition of 5 c.c. of glycerol, and 2·5 c.c. of 50% potassium hydroxide are saponified, and the Reichert–Meissl and Polenske values determined. The titrated distillate from the Reichert determination is diluted with water to 130 c.c. and 0·5 g. of finely powdered silver sulphate is added. After keeping for 1 hr. with occasional shaking the mixture is filtered and 120 c.c. are distilled after the addition of 25 c.c. of 2·5% sulphuric acid. 110 c.c. of the distillate are titrated with 0·1N-soda; the result multiplied by 1·2 gives the Kirschner value. By means of formulæ and a table the percentages of milk fat and coconut oil are obtained. Methods are also given for the examination of sweets containing milk fat. For ash, 5—10 g. of an average sample are incinerated in the usual way; if the ash is less than 0·6%, the sample is doubtful, if less than 0·45 there is an insufficiency of milk. Albumin is determined by dissolving 5 g. in hot water, precipitating with copper sulphate solution, filtering through asbestos, washing, drying with alcohol and ether, weighing, igniting, and weighing again. The normal amount of albumin in milk sweets is about 2·5%. Fat is determined on 5—10 g., after precipitating the albumin with copper sulphate, by the Röse–Gottlieb or the Kuhlmann and Grossfeld process, and for further examination the saponification value, and the Reichert, Polenske, and Kirschner values are determined.

W. G. CAREY.

Relations between the constants of fats. H. WOLFF (Z. angew. Chem., 1926, **39**, 1037—1038; cf. Schay, B., 1926, 593).—The formula $n = 1 + d(0·5557 - 0·00022V + 0·000035I)$, where n is the refractive index, d the density measured at the same temperature, V the saponification value, and I the iodine value, deduced on theoretical grounds, is confirmed completely by actual determinations.

S. I. LEVY.

Fluorescence of oils in ultra-violet light. F. CRONER (Z. angew. Chem., 1926, **39**, 1032).—The fluorescences observed with various fatty and mineral oils both before and after heating to specific temperatures, are recorded. A dark blue fluorescence at the surface indicates a fatty oil which has been heated above 150° or a mixture of fatty oil and mineral oil. A mixed colour indicates a mixture of different fatty oils.

S. I. LEVY.

Detection of unsaponifiable oils in fats. D. HOLDE and A. GORGAS (Chem. Umschau, 1926, **33**, 198).—Unsaponifiable matter frequently cannot be detected in marine animal oils by the ordinary qualitative test, although present to the extent of 10%. By the addition of cold water, drop by drop, or of a larger quantity (10 c.c.) of hot water to the saponified test mixture, the unsaponifiable constituent can be detected. Small amounts of various unsaponifiable oils when added to linseed or cottonseed oils can be detected in a similar manner. Surface tension appears to be an important factor in the detection of unsaponifiable matter in fats.

F. R. ENNOS.

Additive products of iodine monobromide and hypoiodous acid with unsaturated compounds. D. HOLDE and A. GORGAS (Chem. Umschau, 1926, **33**, 198).—Calcium salts of the bromoiodo- and iodohydroxy-fatty acids previously described (B., 1925, 600) were prepared. Bromoiodohexane was obtained from methylpropylethylene by the action of Hanus' solution as a slightly coloured liquid, b.p. 124—126°, f.p. —18°.

F. R. ENNOS.

Deodorisation of coconut oil. W. L. BROOKE (Philippine J. Sci., 1926, **30**, 201—212).—The author confirms the finding of Haller and Lassieur (B., 1910, 704) that methyl nonyl ketone is present in the sludge obtained in the deodorisation of coconut oil. Alcohols are also present. Most of the unsaponifiable constituents of the oil distil over during the first four hours of deodorisation, but in the factory process only about a tenth of the yield is recovered, the remainder being removed in the water used to maintain the vacuum.

C. P. STEWART.

Distillation of fatty oils in a vacuum. D. HOLDE and A. GORGAS (Chem. Umschau, 1926, **33**, 197—198).—The residue from the distillation of linseed oil *in vacuo*, when mixed with Para rubber and vulcanised, does not make so good a rubber substitute as the ordinary factis. The residue from the distillation at 20 cm. pressure is a dark, viscous liquid. The semi-solid distillate (14—19% of the linseed oil) consists chiefly of saturated acids from which stearic acid can be separated, together with 13% of unsaponifiable oils, mainly unsaturated hydrocarbons with small quantities of aldehydes, ketones, and alcohols. Distillation of cod-liver oil similarly gives 31—38% of distillate, consisting of unsaponifiable oils and saturated acids, chiefly palmitic acid, together with 54—62% of an elastic residue.

F. R. ENNOS.

Polymerisation and oxidation of unsaturated fatty acids. K. H. BAUER (Chem. Umschau, 1926, **33**, 198—199).—When α- and β-elæostearic acids are heated in carbon dioxide to 200°, polymerisation products are obtained having lower acidity, higher saponification values, and lower iodine values than the parent acid. The mol. wt. of the β-polymer is 985·6 as determined by the depression of the freezing point of benzene and 490 by Rast's camphor method, the corresponding figures for the polymer of the α-acid being 4588 and 2285 respectively. Catalytic hydrogenation of the α-polymer yields chiefly stearic acid, but the β-polymer gives a thick oil of mol. wt. 1030 in benzene and 535 in camphor. The fatty acids from polymerised linseed oil give mol. wt.

values which show that the product is bimolecular in benzene solutions and unimolecular in camphor solutions, thus differing from the fatty acids from polymerised perilla oil (B., 1925, 250). On oxidation with perbenzoic acid, linolenic acid takes up 4 atoms of oxygen and its ethyl ester 3. Oleic acid similarly takes up 2 atoms of oxygen and its ethyl ester one only. The oxidation products of the acids are approximately bimolecular in solution, but those of the esters are unimolecular (cf. B., 1925, 459). F. R. Ennos.

Synthesis of waxes. A. Grün (Z. angew. Chem., 1926, **39**, 1037; cf. A., 1926, 596).—Good results are obtained in the preparation of wax alcohols from higher ketones by using metals other than nickel, and mixtures of metals, as catalysts, yields of 80—90% being obtained. Combustion of the products in the ordinary way for analysis gives low results, ethylene escaping unburned, but accurate results are obtained if the material is mixed with cupric oxide in the boat. S. I. Levy.

Cotton seed and its products [during the past fifty years]. D. Wesson (Ind. Eng. Chem., 1926, **18**, 938—940).

Progress in the soap industry during the last fifty years. M. H. Ittner (Ind. Eng. Chem., 1926, **18**, 908—910).

See also A., Sept., 938, **Isomerism of dihydroxystearic acids produced by oxidation of acids of the oleic and elaidic series** (Hilditch). 977, **Connexion between degree of dispersion of substrate and enzyme action. Determination of enzymic lipolysis** (Rona and Kleinmann). 980, **Unsaponifiable fraction of fish oil** (Weidemann). 981, ***Ochna pulchra* berries and oil** (Facer); **Flower waxes: rose wax** (Prophète).

Patents.

Food and other products from fish (E. P. 246,148).—See XIX.

Working up slaughterhouse offal etc. (E.P. 256,162).—See XIX.

Substances for salves (G.P. 425,331).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Examination of kauri dust. H. Wolff (Farben-Ztg., 1926, **31**, 2609—2611).—When the very divergent analytical constants determined on various samples of kauri dust are calculated to values for the portion soluble in amyl alcohol, improved concordance is obtained, and in particular the sum of the saponif. value and iodine value is nearly constant (340—370 being the suggested limits). Formation of phenolic hydroxy-groups by oxidation at the double linkings is suggested as a possible explanation for this reciprocal fluctuation in saponif. and iodine values, which had previously been observed in the case of lump kauri. Examined over a period of 20 years, a sample of kauri dust showed a gradual increase in saponif. value, while its acid value and iodine value dropped towards a minimum. The sum of saponif. value and iodine value remained approximately constant (335—350) for 13 years, but then rose with the saponif. value, attaining a value of 385 at the end of the observations. The solubility dropped by 9% during the whole period. The determination of the amount and nature of the ash in the insoluble matter gives a possible further criterion that, taken in conjunction with the above considerations, enables adulteration (particularly with rosin) to be detected. S. S. Woolf.

Storch–Morawski [Liebermann] reaction and rosinate-varnishes. H. Wolff (Farben-Ztg., 1926, **31**, 2611—2612).—A critical discussion of Schulz and Krämer's work (B., 1926, 796). The limitations of the Storch–Morawski reaction for rosin are pointed out, with special reference to its application to the specifying of varnishes as rosin-free. S. S. Woolf.

Paint and varnish—yesterday, to-day, and to-morrow. M. Toch (Ind. Eng. Chem., 1926, **18**, 948—949).

Acid constituents of resin of *Pinus pinea*. Dupont and Dubourg.—See A., Sept., 954.

Patents.

Manufacture of lampblack. Verein für Chem. & Metall. Produktion (G.P. 429,485, 17.10.23).—The usual starting materials (except methane, ethylene, and gases containing these) are mixed with hydrogen before combustion. Materials of higher oxygen content require more hydrogen than those of smaller oxygen content. A. Davidson.

Lining and coating pipes and other bodies. B. Talbot (E.P. 255,546, 24.4.25).—An improved coating material for applying to the interiors of pipes etc., centrifugally or otherwise, consists of bitumen mixed with inert filling material and with 5—10% of sulphur, and heated for several hours at a temperature above the melting-point of the bitumen, until evolution of gas has ceased. The inert powdered loading material may be added before or after the treatment with sulphur. The sulphur appears to combine with gases in the bitumen, as hydrogen sulphide is largely evolved, and but little sulphur remains combined with the bitumen. A bitumen already containing about 5% of combined sulphur was greatly improved (melting-point raised and rendered less brittle when cold) by treatment with a further 5% of elemental sulphur. B. M. Venables.

Manufacture of paints and the like. W. E. Billinghame (E.P. 255,911, 23.1.25).—Bitumen or bituminous material is emulsified in admixture with pigments. Alternatively, an emulsion of the vehicle may be added to the pigment mixed with an alkaline solution of a soluble protein or to an emulsion of the pigment ground in oil. (Reference is directed, in pursuance of Sect. 7 (4) of the Patents and Designs Acts, 1907 and 1919, to E.P. 221,380, 154,236, 15,711 of 1910, and 11,208 of 1903.) E. H. Sharples.

Purifying and stabilising hydrocarbons [turpentine oil]. F. Klein (U.S.P. 1,592,603, 13.7.26. Appl., 19.8.25).—Oil of turpentine is fractionally distilled and the 150—165° fraction is successively heated to

156° and cooled to 20° in one container, until no more of the liquid distils. The yield of terpene boiling at 155—156° is greater by 30—40% than that obtained by the usual method of fractional distillation. The method can be applied to the fractional distillation of a number of hydrocarbon mixtures. T. S. WHEELER.

Condensation products of phenol and the like. A. H. BROWN, and SILUMINITE INSULATOR CO., LTD. (E.P. 255,516, 17.4.25).—If to cold well-stirred phenol–formaldehyde or similar mixtures yielding resinous condensation products, with or without accelerators, there is added hot linseed, castor, or other suitable water-immiscible oil (in amount not exceeding 3·2% by volume of the remaining ingredients), a separation into two layers occurs on keeping, the whole of the water present sinking to the bottom. The upper oily layer, containing the intermediate reaction product, is run off and heated, when it will thicken to any desired extent and the condensation reaction will not proceed beyond the stage attained when the heating is stopped. The addition of commercial phenol will reduce the hardness of the mass if the reaction has been carried too far. S. S. WOOLF.

Manufacture of artificial resins. R. SINGER, Assr. to KUNSTHARZFABR. REGAL & CO. (U.S.P. 1,590,961, 29.6.26. Appl., 17.6.25. Conv., 19.5.25).—7 pts. of monochloro- or dichloro-aminoaldehyde are dissolved in 80 pts. of 40% formaldehyde and 100 pts. of phenol; the mixture is boiled under a reflux and then heated under reduced pressure to remove water. A clear resin is obtained. T. S. WHEELER.

Dehydrated carbohydrate–phenolic resinous products. J. V. MEIGS (U.S.P. 1,593,342, 20.7.26. Appl., 30.11.25).—600 pts. of phenol, 5 pts. of sulphuric acid, and 500 pts. of sugar containing 84% of dextrose are heated to 180°, the water formed being removed through a fractionating column. 8 pts. of stearic acid are added as a lubricant for moulding purposes, and the excess of phenol is then removed by heating *in vacuo*. A black resin is obtained, which appears from the quantities of phenol and dextrose entering into reaction and the quantity of water produced, to be formed by interaction of 1 mol. of dextrose and 2 mols. of phenol. The resin melts to a viscous liquid and may be hardened by heating to 140° with 3—10% of hexamethylenetetramine. In place of phenol, α- and β-naphthol, aniline, and α- and β-naphthylamine may be used, whilst dextrose may be replaced by other hexoses or by pentoses. T. S. WHEELER.

Production of oven-drying lacquers. CELLA DRAHTWERK G.M.B.H. (G.P. 428,390, 25.3.24).—Colloidal palladium accelerates the drying of lacquers. An addition of 5 g. of palladium to 50 kg. of lacquer enables drying to take place at 100° in the same time as is required at 200—230° without palladium. A. DAVIDSON.

Refining aldehyde resins. W. O. HERRMANN and H. DEUTSCH, Assrs. to CONSORTIUM FÜR ELEKTROCHEM. IND. G.M.B.H. (U.S.P. 1,596,971, 24.8.26. Appl., 15.6.22. Conv., 29.6.21).—See E.P. 182,459 and 184,442; B., 1923, 613 A.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Artificial ageing tests on plantation rubber. LONDON COMMITTEE, CEYLON RUBBER RESEARCH SCHEME (Bull. Imp. Inst., 1926, **24**, 209—219).—When vulcanised with sulphur only, unsmoked sheet rubber is superior to crêpe rubber in tensile strength, in latitude of vulcanisation, and in ageing properties; it is, however, less plastic and more variable in rate of vulcanisation than crêpe rubber. Pressing the crêpe rubber into blocks and rolling up the sheet rubber with retention of a proportion of moisture has no effect on the ageing properties. D. F. TWISS.

Preservatives against ageing for rubber vulcanised with sulphur chloride. W. ESCH (Gummi-Ztg., 1926, **40**, 2536—2537).—A review of the precautions necessary and of the preservatives favourable to the production of stable cold-vulcanised products. The preservatives, such as aniline, naphthol, and aldol-α-naphthylamine, are applied in solution after the vulcanisation process. D. F. TWISS.

Sols of caoutchouc nitrosite-nitrosate. K. FISCHER (Gummi-Ztg., 1926, **40**, 2587—2588).—Solutions of several samples of rubber in nitric acid were examined as to their viscosity and surface tension on the addition of various proportions of water and were shown to be highly dispersed sols; the physical character of the nitrosite-nitrosate precipitable by more water was also investigated as to its physical properties and colloidal character. D. F. TWISS.

Changes in the rubber industry during the past fifty years. G. OENSLAGER (Ind. Eng. Chem., 1926, **18**, 902—905).

PATENTS.

Manufacture of coloured rubber goods. I. G. FARBENIND. A.-G., Assees. of W. ZIESER (G.P. [A] 427,873, 20.3.21, and [B] 428,687, 24.9.21).—(A) Mixtures of rubber with organic colours, if also containing zinc oxide, a piperidinecarbothionolate, and a reduced proportion of sulphur, can be vulcanised at low temperatures without loss of brilliancy. (B) Mixtures containing inorganic pigments sensitive to the conditions of ordinary vulcanisation can in a similar manner be vulcanised below 100°. D. F. TWISS.

XV.—LEATHER; GLUE.

Tannins of the black cypress pine (*Callitris calcarata*) and their distribution in the bark. F. A. COOMBS, W. MCGLYNN, and M. B. WELCH (J. Proc. Roy. Soc. N.S.W., 1926, **59**, 356—382).—Black cypress pine, *Callitris calcarata*, yields an important tannin-bearing bark available in large quantities. The tannin occurs principally in the phloem parenchyma, the medullary ray cells, and the epithelial cells lining the resin passages. Some specimens contain nearly 37% of tannin, and commercial samples average 20—25%. For commercial extraction the bark should be finely ground, and a moderate temperature employed, since there is a loss of tannin owing to the probable formation of starch–tannin complexes at higher temperatures. C. J. STILL.

Use of filtered ultra-violet light for recognising and distinguishing natural and artificial tanning materials. O. Gerngross, N. Bán, and G. Sándor (Z. angew. Chem., 1926, 39, 1028—1032).—Nearly all the modern synthetic tanning preparations show fluorescence when examined by ultra-violet light from a quartz mercury lamp passed through a dark glass filter, which cuts off all the visible rays. Tables of colours are given for 24 fluorescent materials, most of which answer the test in dilutions of 1 : 1,000,000. A list of 14 materials which give no fluorescence at dilutions of 1 : 1000 is also given. The fluorescence exhibited by natural tannins under the same conditions is very different; with these, in many cases, there is local absorption, and they yield the fluorescent components to fibres immersed in the solutions. The violet fluorescence of unbleached sulphite-cellulose is found to be due to an original constituent of the spruce bark, which may be extracted by digestion with pure water at 120°. S. I. Levy.

Behaviour of neutral salt-treated hide powder towards tanning agents. K. H. Gustavson (J. Amer. Leather Chem. Assoc., 1926, 21, 366—385).—Portions of hide powder were treated with molar solutions of various salts and the loss of hide substance was determined. The peptising action of cations was shown to be in the order $Ca>Sr>Ba>Mg>Na$, K, and that of anions $CNS>I>Br>Cl>SO_4$, S_2O_3. Portions of hide powder treated respectively with different neutral salts were treated with a solution of hemlock extract, and the combined tannin was determined in each case by the Wilson–Kern method. The p_H values of the exhausted tan liquors were the same in every case, but the combined tannin was greatest in the case of those powders which had lost most hide substance by peptisation, *i.e.*, it followed the Hofmeister series. The author considers that the combination of vegetable tannin with collagen is largely a function of the state of the hide powder in regard to its secondary valency activity and specific surface forces. Vegetable tanning cannot be explained simply as an electro-neutralisation process. Cathodic chromium in the form of chlorides and sulphates gave the same chromium fixation for all the samples of hide powder, whereas the fixation of anodic chromium depended on the previous treatment of the hide powder and followed the Hofmeister series. Extremely basic chromium sulphate liquors containing both cathodic and anodic chromium showed specific ion-effects in their reactions with hide powder treated with neutral salts. In this case it is suggested that there is primary valency reaction between cationic chromium and the carboxyl groups of the protein, formation of molecular compounds between basic protein groups and anionic chromium complexes, and true adsorption of chromium salts by the hide powder. Hide powders treated with neutral salts behaved towards basic aluminium sulphate in the same way as towards extremely basic chromium compounds. D. Woodroffe.

Determination of moisture in tannery materials. I. D. Clarke (J. Amer. Leather Chem. Assoc., 1926, 21, 385—399).—The Bidwell–Sterling modification of the Dean–Stark method (cf. B., 1925, 268) can be applied to all the substances which offer difficulties by the ordinary method. The sample and 80—100 c.c. of toluene are placed in an Erlenmeyer flask fitted with a delivery tube, which is attached to the upper portion of a measuring tube fitted with a reflux condenser. In this way a reasonably small volume of toluene can be used. Drops of water on the sides of the measuring tube are dislodged by a thin wire loop, whilst a brush is used for removing water from the condenser to the collecting tube. The condenser and tube should be cleaned each time with chromic acid mixture. Comparative determinations have been made using benzene, toluene, and xylene respectively, also with the direct flame method, oven drying, and evaporator and dryer method. The results with benzene, toluene, and xylene did not always agree. The simple xylene distillation method gave lower results than the Bidwell–Sterling modification or the direct flame method. The latter gave results but little higher than those with toluene. Toluene seems to be the most suitable liquid to use. Xylene is unsatisfactory in the case of substances which are liable to decompose. Benzene gives very low results. D. Woodroffe.

Properties of shoe leather. VI. Resilience. J. A. Wilson and E. J. Kern (J. Amer. Leather Chem. Assoc., 1926, 21, 399—403).—The resilience of the 18 different leathers previously mentioned (B., 1926, 504, 600, 798) has been determined. Semichrome leather had the lowest resilience and vegetable-tanned sole leather the greatest. Increasing water content of leather lowers its resilience. D. Woodroffe.

Tanning, 1876—1926. J. A. Wilson (Ind. Eng. Chem., 1926, 18, 934—936).

Artificial leather [during the past fifty years]. G. C. Given (Ind. Eng. Chem., 1926, 18, 957—958).

Patents

Treatment of hides and skins prior to tanning. J. Hell (E.P. 255,566, 1.5.25).—Raw hides and skins, with or without a preliminary soaking in water, are soaked in sulphite-cellulose waste liquor or products recovered therefrom, and the excess sulphite-cellulose material is removed by washing. The hides or skins are then limed, delimed, and tanned as usual. The deliming or pickling may be carried out in the presence of sulphite-cellulose waste liquor or products recovered therefrom and auxiliary agents for plumping the hides may be added. D. Woodroffe.

Manufacture of dry products [bates] from pancreas. R. Lepetit (U.S.P. 1,590,388, 29.6.26. Appl., 22.9.22. Conv., 12.11.21).—Finely-divided pancreas gland tissue is ground with one or more anhydrous inorganic salts which form crystals containing water of crystallisation, an ammonium salt, and sodium chloride, to give a dry product which is of value as an artificial bate. Suitable anhydrous salts include calcium sulphate, sodium sulphate, and magnesium sulphate. A preferred composition comprises ground pancreas material 15—20 pts., anhydrous calcium sulphate 9 pts., dry sodium chloride 16 pts., anhydrous sodium sulphate 15 pts., and dry ammonium chloride 15 pts. T. S. Wheeler.

Dyeing of leather. M. C. Lamb (E.P. 255,555, 27.4.25).—Vegetable-tanned, mineral-tanned, oil-dressed,

or aldehyde-tanned leathers are treated with 1—3% of a hydrogenated naphthalene, *e.g.*, tetrahydronaphthalene, alone or in association with halogen-hydrocarbons and with or without emulsifying agents such as sodium, potassium, or ammonium ricinoleate, sulphonated or solubilised oils of mineral, vegetable, or animal origin, hydrogenated phenol soap, hydrogenated cresol soap, or salts of aromatic acids or of aromatic sulphonic acids, and afterwards dyed in the usual manner. The hydrogenated naphthalene may be mixed with halogen-hydrocarbons and a suitable oil or fatty matter for the purpose of fat-liquoring the leather subsequent to the dyeing process. D. WOODROFFE.

Treating organic material. [Production of artificial leather.] E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,602, 29.6.26. Appl., 17.6.24).—A sheet of cellulose is passed over a felt-covered roller, partly immersed in water, so that the under surface of the sheet is moistened. The upper surface is then treated with a solution of cuprammonia which penetrates the sheet but gradually loses its solvent power as it meets the water. The sheet then passes into a closed electrolytic tank, containing dilute ammonium nitrate, between copper electrodes, where it loses the solvent constituents. The sheet is washed, dyed while still moist, and passed over hot rollers, where the treated surface is stamped to simulate leather. R. B. CLARKE.

Preparation of plastic horny substances from albumin or albuminous materials. DEUTSCHE KUNSTHORN-GES. M.B.H., and J. SCHLINCK (G.P. 429,525, 21.10.24).—The materials are treated with a dilute solution of formamide, acetamide, or mixtures of the two either before or after the plastifying process. The hardening effect is slow and does not interfere with the plastifying process. C. IRWIN.

Treatment of cellulose (U.S.P. 1,590,607).—See V.

Food and other products from fish (E.P. 246,148). —See XIX.

XVI.—AGRICULTURE.

Composition of the fractions separated by mechanical analysis from some Transvaal soils. B. DE C. MARCHAND and C. R. VAN DER MERWE (S. Afr. J. Sci., 1925, **22**, 104—118; Chem. Abstr., 1926, **20**, 1880). —In sandy soils the silica decreases, whilst alumina, ferric oxide, phosphoric oxide, lime, magnesia, and potash increase with decrease in the size of the soil aggregate. The phosphorus and potash are largely concentrated in the clay. In the red clay or heavy red loam soils the silica decreases, the alumina, magnesia, and phosphate increase, with decrease in particle size, but the other constituents exhibit irregularities. Considerable proportions of phosphorus and potash are present in the clay fractions. In the black clay soils silica and lime decrease; alumina, ferric oxide, magnesia, potash, and phosphate tend to increase from the coarser to the finer fractions, the potash and phosphate again being concentrated chiefly in the clay fraction. It is noteworthy that in the black clay soils the concentration of silica is relatively high in comparison with that of alumina, whilst the reverse is the case for the red clays. The clay fraction of sandy soils is similar in composition to that of the heavy red soils and different from that of the black. A. A. ELDRIDGE.

Decomposition of organic matter in soil. H. H. HILL (J. Agric. Res., 1926, **33**, 77—99).—Pure cellulose, whether added to soil or to culture solutions in which plants were grown, restricted plant growth to an extent proportional to the amount of cellulose added. Treatment with 0·01% of potassium nitrate did not counteract this effect. During the cellulose decomposition hydrogen sulphide was produced by the reduction of sulphates in the culture solution. The root systems of plants grown in culture solutions containing cellulose were much discoloured and unhealthy. Maize and wheat following ploughed-in legumes showed increased yields, but wheat following ploughed-in rye-grass showed decreased yields compared with controls over a twelve-year period. The nitrogen–carbon ratio of the green crop was higher for non-legumes than for legumes, and generally the ratio was smaller in younger plants. The evolution of carbon dioxide following the incorporation of green matter with soil was greater than when hayed crops were used. In similar circumstances nitrate production was more rapid from air-dried material than from green, and was considerably greater with legumes than with non-legumes. A. G. POLLARD.

Arsenates as soil insecticides. B. R. LEACH (J. Agric. Res., 1926, **33**, 1—8).—Acid lead arsenate proved toxic to the larvæ of the Japanese beetle (*Popillia japonica*) when mixed with soil in which the larvæ were feeding. The death period varied with the amount of the arsenate added. The arsenates of lead (basic), iron, and magnesium were non-toxic under these circumstances. These arsenates were also non-toxic to plants. Acid lead arsenate was the least toxic to plants among those arsenates toxic to the larva (zinc, copper, and calcium arsenates were also examined). In the soil acid lead arsenate appears to be converted slowly into a basic form, but can be applied at the rate of 1500—2000 lb. per acre in nursery soils without affecting the growth of plants. A. G. POLLARD.

Relative availability of phosphatic fertilisers on acid and non-acid soils. G. INGHAM (S. Afr. J. Sci., 1925, **22**, 122—134; Chem. Abstr., 1926, **20**, 1882).—The "availability" (subsequent solubility in 1% citric acid) of a phosphatic fertiliser depends more on the soil reaction than on the water- or citric acid-solubility of the original fertiliser, varying for superphosphate from 21% in a very acid soil to 96% in a soil free from acidity. The availability is closely related to the iron, aluminium, and organic matter content of the soil. Low availability may be due to the absorptive power of organic and inorganic colloids, especially those of iron and aluminium. Acid soils have a high absorptive capacity for phosphate as well as for lime. Removal of calcium carbonate from a soil containing much iron and aluminium doubled the absorption capacity and reduced the citric acid-solubility of the fertiliser by 50%, whereas addition of calcium carbonate to an acid soil increased the availability

of superphosphate only after 12 months. The use of carbonic instead of citric acid would probably be in closer accord with soil conditions. A. A. Eldridge.

Phosphates and phosphoric acid requirements of soils. M. von Wrangell (Landw. Jahrb., 1926, **63,** 627—668; Chem. Zentr., 1926, II., 815—816; cf. succeeding abstracts).—The concentration of phosphoric acid in the soil solution depends not only on the amount of water, but also on the presence or absence of other ions; further, it depends more upon the absorptive capacity of the soil than upon the chemical nature of the phosphate. It is markedly influenced by the presence of lime, or a change in the reaction of the soil. The amount varies from 0·02—2·0 mg. per litre in different soil types, with an average figure of 0·1—0·6 mg. The speed with which equilibrium between soil and soil solution is restored after disturbance, as by dilution or the action of plant roots, is also an important factor in connexion with the supply of phosphoric acid; it is much less in heavy, strongly absorptive soils than in light soils. The total amount of soluble P_2O_5 in the soil is probably of greater importance than the concentration of the soil solution, since plant roots and root-hairs not only draw on the soil water, but can also obtain P_2O_5 from the colloid coating of the soil particles. The determination may be made by successive extractions of a small amount of soil with distilled water, until a constant minimum concentration is reached, usually about 0·1 mg. P_2O_5 per litre. In practice, the total amount of extractable P_2O_5 may be calculated from the results of the first two or three extractions. Experiments with plants in continuously renewed solutions show that the concentration at which there is no further uptake of P_2O_5 is below 0·1 mg. per litre—usually 0·03 mg. The sap of plants may contain up to 500 mg. per litre of dissolved P_2O_5, which is probably combined with protein. A transference of phosphate ions into colloidal solution or a greater solubility of P_2O_5 in the plasma than in water must be assumed.

C. T. Gimingham.

Colorimetric method for rapid determination of phosphoric acid in very dilute solutions [soil extracts etc.]. M. von Wrangell (Landw. Jahrb., 1926, **63,** 669—675; Chem. Zentr., 1926, II., 816).—90 c.c. of the solution, containing 0·01 to 0·04 mg. P_2O_5, are treated with 1·5 c.c. of the molybdate reagent (equal parts of concentrated sulphuric acid and 10% ammonium molybdate solution), 0·75 c.c. of freshly-prepared 1% stannous chloride solution is added, and the whole made up to 100 c.c. and well shaken. A blue colour appears and reaches its maximum intensity in 5 min. It is compared in a colorimeter with standard solutions. Oxidising substances must be absent; the solution must not be filtered, but may be centrifuged, if necessary. The colour is increased by the presence of arsenic or silica, and reduced by fluorine compounds. With soils, the error due to these is usually negligible, though arsenic should be tested for. If the method is used for "Rhenania" phosphate, serious errors are introduced unless the silica is first removed.

C. T. Gimingham.

Solubility relationships in their application to tertiary phosphates. M. von Wrangell and E. Koch (Landw. Jahrb., 1926, **63,** 677—706; Chem. Zentr., 1926, II., 817).—The action of water on tertiary phosphates is a process of decomposition rather than of solution. In solubility experiments with tertiary phosphates of calcium, aluminium, and iron, equilibrium between the solid and liquid phases could hardly be reached, whereas with crystalline magnesium phosphate, $Mg_3(PO_4)_2,22H_2O$, it was obtained in a short time. By the action of water on calcium phosphate, ions of three kinds are formed—PO_4''', HPO_4'', H_2PO_4'—and a hydrolytic equilibrium is set up; the process of solution alters the acidity of the soil mass, or, conversely, the soil is altered by increase of acid. The more acid the solution, the higher is the content of calcium phosphate, whereas the content of aluminium and iron phosphates increases with increasing concentration of hydroxyl ions; magnesium phosphate is intermediate in this respect. The final result of the hydrolytic decomposition of difficultly soluble phosphates depends considerably on the nature of the base of the phosphate, on the influence of acid on the specific effect of the acid anions, and on the influence of base on the cations. The different effects of sodium and calcium hydroxides are specially striking. Calcium carbonate has a greater inhibiting effect on the solution of tricalcium phosphate than an equivalent amount of calcium chloride. The action is less marked with magnesium, iron, and aluminium phosphates; but soluble calcium salts reduce the PO_4 ions by formation of calcium phosphate. Solutions of ammonium salts exert a much greater solvent action on tricalcium phosphate than solutions of sodium or potassium salts; the effect of the former increases with increasing concentration, whereas that of the latter changes only slightly. Study of the solubility relationships of the phosphates suggests the possibility of controlling the supply of soluble phosphate in the soil by varying the conditions.

C. T. Gimingham.

Phosphoric acid content of natural soil solutions. M. von Wrangell and W. Haase (Landw. Jahrb., 1926, **63,** 707—738; Chem. Zentr., 1926, II., 817—818).—Methods for obtaining the natural soil solution are discussed. Direct displacement of the solution by pressure is the most satisfactory, and details are given of a suitable type of hydraulic press. It is important that the pressure should be great in relation to the height of the column of soil. The slightly turbid liquid obtained is purified by flocculating the colloidal matter present and centrifuging. Phosphoric acid can then be determined by the colorimetric method (cf. preceding abstract). The concentration of P_2O_5 in the soil solution of different soils varies widely, but is relatively constant in any one soil at different times of the year. There appears to be no relation between the amount of P_2O_5 in the soil solution and the total present in the soil. The effect of the treatment of the soil with very dilute acid or alkali on the concentration of P_2O_5 varies greatly with the nature of the soil. With some soils, a second displacement of the solution, after remoistening of the soil, shows about the same concentration of P_2O_5

as the first; with other soils there is a marked decrease. C. T. GIMINGHAM.

"Available" part of the phosphoric acid in soil. M. VON WRANGELL and L. MEYER (Landw. Jahrb., 1926, **63**, 739—775; Chem. Zentr., 1926, II., 818—819; cf. preceding abstracts).—Neubauer's seedling method for determining the P_2O_5 requirement of soils is critically compared with von Wrangell's method of determining P_2O_5 colorimetrically in the natural soil solution displaced by pressure. The value obtained by the former method is liable to vary from time to time under the influence of changing conditions, whereas the latter determines a relatively fixed quantity. Careful control of the quality of seed, the temperature, the water content of the soil and other factors is most important in carrying out the Neubauer method. Comparative experiments with both methods indicate a relationship between the availability of the soil P_2O_5 and the concentration in the soil solution. With increasing water content, the P_2O_5 concentration of the soil solution increases; on drying, there is a rapid decrease of concentration; and on remoistening, the original figure is reached again in a short time. These relationships are less evident with heavy soils on account of the slow rate at which equilibrium between soil and soil solution is restored after disturbance. The seedling method also shows an increasing uptake of P_2O_5 by the plants, with increased water content of the soil. Addition of sand to the soil decreases the P_2O_5 content of the soil solution, unless the water content is made up to the original figure. It is suggested that the increase of concentration of P_2O_5 in the soil solution when the amount of water in the soil is increased is due to the solution of certain forms of phosphate, probably held absorbed, owing to a change in the mechanical structure of the finest soil particles. C. T. GIMINGHAM.

Nitrogen availability studies on crops harvested at different stages of growth. C. H. SINGLETON (New Jersey Agric. Exp. Sta. Bull., 1925, [421], 1—28; Chem. Abstr., 1926, **20**, 2040).—For oats, millet, buckwheat, and rape, the yields of dry matter after fertilisation were in the decreasing order: sodium nitrate, ammonium sulphate, tankage, calcium cyanamide. The recovery of nitrogen was respectively 62%, 54·24%, 46·62%, 25·84%. The percentage of nitrogen is highest in plants during the first 3 weeks of growth. A. A. ELDRIDGE.

Sulphur as a plant food. J. R. NELLER (Washington Agric. Exp. Sta. Bull., 1924, [187], 37—38; Chem. Abstr., 1926, **20**, 1881).—Comparatively large quantities of sulphur may be oxidised in E. Washington soils without marked retardation of organic oxidation; the addition of large amounts of sulphur to fine sandy loam caused an increase in the water-soluble potassium and calcium. Legumes, in increased yield, frequently showed an increase in total protein, phosphorus, and potassium, and a decrease in calcium. Straw humification proceeds more rapidly on the addition of sodium nitrate or ammonium sulphate and calcium carbonate. The initial stages of decay appear to be associated with the formation of a toxic substance which is subsequently oxidised to a harmless or beneficial compound. A. A. ELDRIDGE.

Effect of nutrient conditions on the colloidal properties of certain vegetable crops. J. W CRIST (Michigan Agric. Exp. Sta. Tech. Bull., 1926, [74], 1—27).—Lettuce, spinach, radish, beet, and tomatoes, when grown on suitably fertilised soil, had a higher ratio of green to dry weight than unfertilised plants. The treated plants had higher percentages of bound water and the hydratable components of their juices were of greater hydration capacity, nitrates being most effective. The efficiency of nitrate is reduced by potassium or phosphorus, but not by sulphur. The relation of the results to the structure and behaviour of the plants is discussed. A. A. ELDRIDGE.

Relationship between concentration of soil solution and physico-chemical properties of leaf-tissue fluids of Egyptian and Upland cotton. J. A. HARRIS (J. Agric. Res., 1926, **32**, 605—647; cf. Harris and others, B., 1924, 607; 1925, 35).—One variety of Egyptian and two varieties of Upland cotton were grown under irrigation conditions in Arizona, the planting being so arranged that sub-plots of the three varieties were replicated many times in the experimental area. Statistical methods of treatment are applied to the data obtained by determinations of (*a*) the concentration of the soil solution (as measured in terms of the electrical resistance of the saturated soil mass) of a large number of samples of soil, taken at depths of 1—4 ft. throughout the field, and (*b*) of the osmotic concentration (freezing point depression), specific electrical conductivity, and chloride content of the leaf-tissue fluids of the plants on each sub-plot. The results show the existence of marked heterogeneity of the soil in a small experimental field and demonstrate the possibility of determining, with considerable precision, the correlation coefficient of the relationship between the properties of the soil of different portions of the field and the characteristics of the plants grown thereon. Differences in the plant tissue fluids are associated with the soil differences, though there is not an exact parallelism. In general, higher concentrations of all solutes, and of dissociated electrolytes in the plant fluids, are associated with higher concentrations of the soil solution. There is also a substantial correlation (especially marked for Egyptian cotton) between resistance of the soil mass and the chloride content of the leaf fluids. The method of treatment could be extended to other properties of soils and crops, and the results are considered to indicate the possibility of carrying out physiological investigations in the field. C. T. GIMINGHAM.

Fertilising in relation to the disease resistance of crops. B. THOMAS (Fert. Feeding-stuffs J., 1925, **10**, 478—488).—Potassium salts, particularly the chloride or sulphate (*e.g.*, the latter at 200 lb. per acre), considerably increase the percentage of healthy potato plants; superphosphate (600 lb. per acre) applied to a soil already containing an unusually large quantity of available phosphate considerably decreased the percentage. Ammonium sulphate supplied up to 800 lb. per acre as a balanced fertiliser with potash and superphosphate afforded a progressive increase in the percentage. Potassium is the determining factor in promoting the disease resistance of plants; nitrogen and phosphate in exces-

sive amounts have an adverse effect. There is an optimum ratio for each variety of plant.

A. A. Eldridge.

Chemistry's contributions to the fertiliser industry [in the past fifty years]. J. E. Breckenridge (Ind. Eng. Chem., 1926, **18**, 941—943).

Insecticides and fungicides [during the past fifty years]. E. C. Holton (Ind. Eng. Chem., 1926, **18**, 931—933).

Losses of nitrogen caused by soil bacteria in pure cultures. Lemoigne and Dopter.—See A., Sept., 979.

Variation in moisture and nitrogen content of potato during growth. Fagan.—See XIX.

Effect of hydrogen-ion concentration of soil on formation and composition of essential oil of wood sage. Deel.—See XX.

Patents.

Manufacture of mixed fertilisers. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 255,726, 7.1.26).—As an improvement on E.P. 236,494 according to which a mixed fertiliser is prepared by mixing together urea, ammonium phosphate, and potassium nitrate and/or sulphate, it is now proposed to replace the ammonium phosphate wholly or partially by dicalcium phosphate. It is claimed that the latter compound is of value on account of its buffer action. Potassium may be used in the form of any fertilising salt but the sulphate and/or nitrate are preferable. (Cf. B, 1925, 732.)

G. W. Robinson.

Decompostion of raw phosphates. Landwirtschaftliche Warenzentrale A.-G. (G.P. 430,095, 4.7.25).—The decomposition of crude phosphates by means of sulphur dioxide is facilitated by the addition of an equivalent, or smaller amount, of chloride, nitrate, oxalate, silicate, permanganate, or persulphate, or mixture of these salts, with or without a sulphate. The introduction of a small quantity of free acid (which must not exceed one-tenth of the phosphoric acid present) is also advantageous.

R. B. Clarke.

Storage of mixed fertilisers containing ammonium nitrate. J. Heppes (G.P. 430,096, 24.12.24).—As usually stored, mixtures of ammonium sulphate and ammonium nitrate, or similar mixtures, set to an extremely hard mass after a short time. This is avoided if the storage heaps are maintained at a temperature above 32°.

R. B. Clarke.

Insecticidal or other toxic substances from plants of the *Derris* species. Nettai Sangyo Kabushiki Kaisha (E.P. 247,140, 25.6.25. Conv., 4.2.25).—Fresh roots of *Tuba* or other of the *Derris* species are crushed, mixed with a small quantity of water, and the mixture is filtered in a centrifugal filter. The liquor is allowed to settle in a tank for about 3 hrs. and the supernatant liquid transferred to a vacuum evaporator. The residues in the tank consist mainly of resinous matter and may be dried separately. The juice is evaporated at a temperature not exceeding 70° and should be constantly agitated to prevent the formation of a greyish, oily skin on the surface. When reduced to a grey-brown paste the contents of the evaporator are spread on glass or iron plates and dried in a drying chamber at 40—45°. The resulting brown powder can be stored indefinitely without deterioration, and on the addition of water produces a milky-white emulsion.

A. G. Pollard.

Method of combating plant diseases. J. D. Riedel A.-G. (G.P. 425,496, 8.5.21).—The plants are sprayed with a solution developing sulphur and sulphur dioxide. A dilute, aqueous solution of sodium thiosulphate acidified, just before use, with 5% hydrochloric acid is suitable. A coating of colloidal sulphur becomes deposited on the plants.

R. B. Clarke.

Material for combating plant diseases and treating seeds. R. Falck (G.P. 425,707, 8.10.22).—A solution especially suitable for disinfecting seeds consists of an alkaline solution of a formaldehyde–phenolic resin incorporated with a copper salt. For instance, such a resin is mixed with a solution of a cupric salt. In the complex formed copper is probably present in the phenolic nucleus as it can be detected only on breaking down the molecule. The product is soluble in ammonia solution and also in fixed alkalis, in contrast to copper hydroxide. Carbon dioxide re-precipitates the compound from such solutions. When a solution in caustic soda is sprayed on to young plants a resinous film, impermeable to rain, is formed. The copper applied in this form exerts a considerably greater destructive action on fungi than a corresponding amount applied as an acid solution of copper sulphate. After destruction of fungi the copper compound is subsequently decomposed by the carbon dioxide of the air. It does not affect injuriously the germinating power of seeds.

R. B. Clarke.

Materials for spray fluids and seed pickling. A. W. Beer (F.P. 580,438, 5.1.23.)—Tanning extracts or protein precipitants with tanning properties are claimed as fungicides; *e.g.*, 0·25—0·5% aqueous solutions of protocatechuic acid, gallic acid, or pyrogallol will kill fungus spores in 10—20 min. Tannins insoluble in water may be dissolved in appropriate organic solvents or made soluble in water by the addition of a little sulphuric acid. The fungicidal action of tannins may be increased by addition of substances which increase their diffusibility, such as mineral or organic acids, ethers, ketones, or salts. Such additions also increase the efficiency of known fungicides; *e.g.*, the time required for a 0·5% solution of copper sulphate to kill "smut" spores is reduced from 15—16 hrs. to 8—10 min. when the solution contains 0·1% of monochloroacetic acid. The wetting power of tannin solutions is improved by addition of phenol, acetin, sulphite-cellulose waste lye, or pentosans. The above-mentioned materials can be used for the protection of seeds against animal pests, such as rodents and birds, if a small amount of naphthenic acid or its salts is added.

C. T. Gimingham.

Increasing the amount and phosphoric acid content of basic slag (G.P. 429,767).—See X.

Food and other products from fish (E.P. 246,148).—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Increase in alkalinity of sugar solutions on evaporation. B. MINAEV (Zapiski [Russia], 1925, **2**, 234—239; Chem. Abstr., 1926, **20**, 2085).—Small amounts of amide-nitrogen present in the beets are decomposed during evaporation; thus conditions favourable for a decrease of alkalinity could not exist. Sulphitation is preferred to carbonation at the third saturation.

A. A. ELDRIDGE.

Increase in alkalinity on evaporation of sugar juices. L. SHAPIRO (Zapiski [Russia], 1925, **3**, 50—54; Chem. Abstr., 1926, **20**, 2085).—Normally the [beet] syrup is unlikely to have the proper reaction after saturation with carbon dioxide at the third saturation; addition of sulphur dioxide is necessary.

A. A. ELDRIDGE.

Determination of ash in sugar manufacture by measuring the electrical conductivity of the aqueous solutions. A. G. ARKHIPOVICH (Zapiski [Russia], 1926, **3**, 95—101; Chem. Abstr., 1926, **20**, 2086).—The conductivity method for the determination of ash, using nickel or platinum electrodes and 2—4 c.c. of a solution of 40° Brix, diluted to 100 c.c., gives satisfactory results.

A. A. ELDRIDGE.

Practical methods of p_H measurement and the scheme of automatic electrometric control of cane juice defecation. R. T. BALCH and H. S. PAINE (Planter and Sugar Mfr., 1925, **75**, 347—350; Chem. Abstr., 1926, **20**, 1915).—Quinhydrone electrodes are unsuitable; for continuous recording a tungsten-manganese sesquioxide electrode is preferred. Charts are given showing changes in p_H for continuously limed defecated juices. The liquid must be cooled and sterile conditions maintained.

A. A. ELDRIDGE.

Honey [analysis]. F. LUCIUS (Z. Unters. Lebensm., 1926, **51**, 351—357).—Reduction or polarisation methods of sugar determination are not satisfactory owing to the presence of other reducing substances, but a separation of sugars from dextrins can be effected by precipitation with ether in alcoholic solution. A mixture of 10 g. of honey and 10 g. of water is treated with 200 c.c. of 96% alcohol, and 100 c.c. of ether are added gradually with continuous agitation. The mixture is left for 24 hrs. and is filtered from the dextrins, which are dried and weighed. The alcohol and ether are evaporated, the residue is made up to 100 c.c., and the sugars are determined by the polarimeter after inversion. In a separate portion the lævulose is decomposed by boiling for 3 hrs. with 20 c.c. of 5*N*-hydrochloric acid and then, after adding alkali till the solution is only slightly acid, the solution is filtered, decolorised with animal charcoal, and examined in the polarimeter.

W. G. CAREY.

See also A., Sept., 941, **Structure of fructose, γ-fructose, and sucrose** (HAWORTH and HIRST); **Constitution of sucrose** (McOWAN).

PATENTS.

Purifying carbohydrate solutions etc. C. B. DAVIS (U.S.P. 1,592,598, 13.7.26. Appl., 26.2.23).—Raw sugar is made into a magma with 10% of water and centrifuged. On diluting the brown syrup thus obtained to *d* 1·075, some of the impurities are precipitated, and after addition of a small quantity of diatomaceous earth and heating to 70°, the solution is filtered through bone char. The resulting colourless liquid is used to dissolve the washed sugar, or other unrefined sugar, until a syrup of *d* 1·263 is obtained, which is purified by treatment with diatomaceous earth, heating to 70°, and filtration through bone char as before. The purified syrup is grained *in vacuo* to obtain a pure sugar.

T. S. WHEELER.

Crystallisation of sugar. C. GRAHAM (Austral. P. 19,812, 29.9.24).—A quantity of sugar juice is introduced into a vacuum pan and boiled until minute crystals of sugar are perceived in a sample of the boiling massecuite when held up before a strong light. The crystals are then caused to grow by running in a syrup which gradually increases in sugar content as the pan fills up. The crystals thus obtained are of uniform size and purity. The essential feature of the process is the blending of the syrups etc. in such a way that the crystals may be caused to grow uniformly.

R. B. CLARKE.

Synthetic bone char (U.S.P. 1,592,599).—See II.

XVIII.—FERMENTATION INDUSTRIES.

Technical control of purchase of barley, malt, and yeast for distilling. S. H. HASTIE (J. Inst. Brew., 1926, **32**, 343—351).—Methods for the evaluation of barley, malt, and yeast for pot-still distillery purposes are indicated, and their value is discussed.

C. RANKEN.

Calculation of percentage of alcohol and other factors in fermented worts. A. SLATOR (J. Inst. Brew., 1926, **32**, 359—360).—A table is given by means of which it is possible to calculate the percentage of alcohol present in beer, the number of degrees of gravity lost, the percentage fermented and other factors, when only the original gravity and attenuation are known. The figures in the table were calculated from the "Mean Brewery Table" of Thorpe and Brown, and are arranged in six columns, recording respectively the degrees of attenuation lost, the degrees of gravity lost per degree of attenuation lost, of alcohol per 100 c.c. per degree of attenuation lost, increase of carbon dioxide concentration per degree lost in attenuation, gravity lost, and increase of alcohol per degree of attenuation lost.

C. RANKEN.

Pectins of grapes and the mellowness of wines. L. SEMICHON and FLANZY (Compt. rend., 1926, **183**, 394—396).—When a must or wine is acidified to the extent of 1% with hydrochloric acid, the addition of alcohol precipitates a mixture of pectins and gums. When the precipitate is dissolved in water the pectic acid separates as calcium pectate and the gums are separated from the filtrate by the addition of alcohol. An examination of a large number of musts and wines has shown that musts contain only pectins, whilst the wines have only gums or mixtures of gums and pectins. It is concluded that grape pectins arise as in other fruit from the decomposition of cellulose tissue, and that the formation of gums during the fermentation of a must is not due to

transformation of the pectins, but that the gums are the products of bacterial action. The bouquet of a matured wine is due largely to the dissociation of the methyl pectic esters or pectins. A. E. Mitchell.

Rapid determination of alcohol in distilled spirits and of colour in whisky. J. F. Williams (Ind. Eng. Chem., 1926, **18**, 841—843).—The alcoholic distillate from wines and cordials and the concentrated distillates from vinegar, ale, cider etc. are extracted with a special reagent consisting of 70 c.c. of amyl alcohol, 28 c.c. of toluene, and 2 c.c. of a 50% aqueous solution of tartaric acid. The extraction is carried out at 20° in a graduated 20-c.c. glass-stoppered cylinder. The height of the lower layer, which consists of the water contained in the distillate, is read and the percentage of alcohol obtained from a table containing the results obtained from a prepared set of 24 standard alcoholic solutions ranging from 5 to 66%. No appreciable interference was noted by the presence of small amounts of glycerol or sucrose. For the determination of colour in the whisky, the cylinder containing the treated spirit is placed against a sheet of white paper, and the approximate percentage of colour in the upper and lower layers noted. So-called natural colour in whisky appears in the upper layer, while any caramel or other water-soluble colours dissolve in the lower or aqueous layers. The test is not a precision method, and is intended for use primarily where a pycnometer and balance are not available, or where the sample is too small for convenient use with these instruments. C. Ranken.

See also A., Sept., 976, **Separation of enzymes of barley malt. Lichenase and cellobiase** (Pringsheim and Beiser); **Protein nature of invertase** (Willstätter). 977, **Formation of polysaccharides by yeast preparations** (Naganishi). 978, **Function of phosphates in dissimilation of hexoses** (Kluyver and Struyk); **Effect of organic substances on glycolysis of yeast** (Schoeller and Gehrke); **Pyruvic acid in alcoholic fermentation** (Traetta-Mosca); **Simpler nitrogenous constituents of yeast. Choline and nicotinic acid** (Vickery); **Determinations of permeability with *Saccharomyces cerevisiæ*** (Söhngen and Wieringa); **Enzymic decomposition in lactic acid bacteria and yeast** (Nilsson and Sandberg); **Acid production of *Aspergillus niger*** (Bernhauer).

Lactic ferment preparations. Placeres.—See XX.

Patents.

Manufacture of pressed yeast. J. I. A. Effront (F.P. 601,484, 18.4.25).—Wort prepared from molasses is purified by treatment with alkali carbonates and nitrogen is passed in during fermentation, so that 30 to 150 mg. of nitrogen are present per litre of wort. The process of assimilation is hastened by leading in very small amounts of oxides of nitrogen during fermentation. C. Ranken.

Bulgarian milk (U.S.P. 1,593,899).—See XIX.

Preparation of citric and other organic acids (G.P. 426,926).—See XX.

XIX.—FOODS.

Determination of protein [in cereals etc.]. H. L. Thompson (Amer. Miller, 1926, **54**, 396; Chem. Abstr., 1926, **20**, 2029).—Wheat proteins (gliadin, glutenin, leucosin, globulin, and proteose) cannot be separated without change in structure. The most accurate factors for calculating total protein from the percentage of nitrogen are: wheat 5·7, rye 5·62, oats 6·31, maize, rice, mixed feed, bran, and "shorts," 6·25. A. A. Eldridge.

Influence of storage on the composition of flour. J. E. Greaves and C. T. Hirst (Utah Agric. Exp. Sta. Tech. Bull., 1925, [194], 1—21; Chem. Abstr., 1926, **20**, 2031).—The milling of wheat in the production of high-grade white flours removes 70% of the ash, 63% of the phosphorus, 90% of the calcium, and 79% of the magnesium. During storage, the soluble carbohydrate and the water-soluble phosphorus increase, whilst the alcohol-soluble phosphorus decreases; the hydrogen-ion concentration first decreases and then increases. No evidence was obtained of protein cleavage during storage. From 1·39 to 3·52% of the nitrogen of these flours occurred as water-soluble basic nitrogen. A. A. Eldridge.

Changes in flour during storage. J. E. Greaves (Amer. Miller, 1926, **54**, 376; Chem. Abstr., 1926, **20**, 2031; cf. preceding abstract).—Only inferior flours greatly increase in acidity during storage for four years. It is concluded that the improvement of the bread-making properties of flours on storage is due to the action of enzymes on the carbohydrates, fats, and proteins. A. A. Eldridge.

Colloid chemistry of wheat gluten. A. Kuhn and G. Richter (Koll. Chem. Beih., 1926, **22**, 421—448; cf. Ostwald, B., 1920, 499 A).—The baking quality of a flour is apparently intimately connected with the nature of the gluten which it contains, and viscosity measurements have therefore been made with sols of the gluten from various types of flour. In every case the viscosity decreases with rising temperature and increasing age of the sol. The gluten from an extract flour (degree of milling 30%) has a higher viscosity than that from a baker's flour (degree of milling 74%) made from the same wheat, the difference depending on the milling. Of the baker's flours examined, the best were those giving the most viscous gluten sols. As the gluten content of a flour decreases with increasing degree of milling, it is necessary, in order to arrive at a correct estimate of the value of the flour, that the sol examined should be of unit concentration. Measurements by Ostwald and Steiner's membrane method (A., 1925, ii, 771) show that the surface tension of the sols decreases with increasing degree of milling, and that the better quality flours give the more elastic sols. R. Cuthill.

Practical method for the determination of the electrical conductivity of milk. V. Gerber (Z. Unters. Lebensm., 1926, **51**, 336—351).—The electrical conductivity is a valuable criterion of the quality of milk, but owing to the cost of the apparatus, as well as the skill and time required, the process is not in general use. The author adopts the method used for determining

the electrical conductivity of other liquids, and an apparatus is described and illustrated in which the ordinary Wheatstone bridge method is used, with a telephone or a galvanometer to determine the position of zero current. Results obtained with many different samples of milk are given. W. G. CAREY.

"Cryolac" number of milk and milk products as a means to determine quantity of added water. P. POST (Pharm. Weekblad, 1926, 63, 983—995).—The name "cryolac" is proposed for the osmotic concentration of lactose and chloride, expressed in Raoult units. The lactose content, chlorine content, and acidity are determined; from the first and last, by reference to prepared tables, the depression of the freezing point which should be caused by the lactose originally present is calculated, and by adding the depression which should be caused by the soluble chloride the cryolac number is obtained. This bears a constant ratio to the total depression, so that the latter may be calculated; figures so obtained for a great variety of samples agree well with figures obtained by actual determination from the milk originally used. The cryolac number of unadulterated milk being taken as 425, the determination enables the proportion of water added to the milk, examined as such or in the form of butter milk, ice cream etc. to be calculated. S. I. LEVY.

Destruction of vitamin-*A* in milk by ultra-violet light. R. W. TITUS, J. S. HUGHES, W. R. HINSHAW, and J. B. FITCH (Ind. Eng. Chem., 1926, 18, 843).—Irradiation of milk, whilst it increases the antirachitic properties (cf. Steenbock, Hart, Hoppert, and Black, A., 1926, 437), simultaneously causes a large decrease in the vitamin-*A* content. J. W. BAKER.

Chemical study of the flesh of emaciated cattle. R. HOAGLAND and W. C. POWICK (J. Agric. Res., 1925, 31, 1001—1013).—Analysis of the flesh of very emaciated cattle indicated a higher moisture content and a lower content of fat, protein, and probably sugar than normal. The average ratio of moisture : protein was greater than 4 : 1, whereas for the flesh from fat cattle the average value was 3·5 : 1. This ratio is suggested as valuable for the purpose of classifying flesh for food purposes. A. G. POLLARD.

Nutritive value of protein in veal and calf sweetbreads; in beef cheek meat, lips, tongues, brains, spleen, and tripe; and in hog brains and tongues. R. HOAGLAND and G. G. SNIDER (J. Agric. Res., 1926, 32, 679—688).—When fed to young rats, in rations containing 10% of protein, for periods of 30 or 60 days, veal, ox brain, tongue, and spleen, hog brain and tongue had approximately the same value as milk as sources of protein for growth and maintenance; ox lips, caseinogen, and beef cheek meat had a somewhat lower value; and sweetbreads and tripe were distinctly inferior. The protein in tripe was insufficient for optimum growth even when fed as 20% of the ration. C. T. GIMINGHAM.

Nutritive value of protein in voluntary muscle, heart, liver, and kidney, from cattle, sheep, and hogs. R. HOAGLAND and G. G. SNIDER (J. Agric. Res., 1926, 32, 1025—1040).—Feeding experiments with young albino rats are reported. The rations contained 10, 12·5, and 15% of protein from the various tissues tested and were prepared so as to have practically the same energy value. The hog tissues (at 10 and 12·5% of protein) had, on the whole, somewhat higher values for maintenance and growth than the others. The differences when 15% of protein was fed were hardly significant. C. T. GIMINGHAM.

Determination of moisture in meat extract. C. DEDLOW and D. T. SMITH (Ind. Eng. Chem., 1926, 18, 858—860).—The usual methods of determining moisture in meat extracts (drying *in vacuo* or in the air oven) yield results which are from 2 to 6% too high, owing to decomposition of the extract at the temperatures used (70° *in vacuo*, 100—105° in the air oven). This degradation continues over a long period, the extract apparently never reaching a constant weight. Improved results are obtained by distillation with toluene under atmospheric pressure and measuring the water carried over, but the most accurate results are obtained by distillation for 2 hrs. at 50° under a pressure of 30—50 mm. with xylene. A detailed description is given of the apparatus used and the procedure followed. B. W. CLARKE.

Vitamin-*A* in poultry flesh and fat. R. HOAGLAND and A. R. LEE (J. Agric. Res., 1926, 33, 29—40).—Nutrition experiments with rats indicated that poultry fat was considerably richer in vitamin-*A* than the flesh. The flesh of chicken, ducks, geese, turkeys, and guinea-fowl was examined, but no definite conclusions as to vitamin content were obtainable. A. G. POLLARD.

Variation in the moisture and nitrogen content of the potato during growth and storage. T. W. FAGAN (Welsh J. Agric., 1925, 1, 110—115).—The moisture content of potatoes gradually decreases during growth, the total nitrogen and protein nitrogen steadily increasing. The ratio of protein nitrogen to total nitrogen also increased to 55—58% at harvest, when the composition was: water, 77·33—78·12; total nitrogen, 0·316—0·297; protein nitrogen, 0·181—0·164%. The leafy portions of the plants exhibited similar variations, the composition at harvest being: water, 22·19—30·32; total nitrogen, 1·149—1·014; protein nitrogen, 0·970—0·812%. During storage the percentages of total nitrogen and dry matter slightly increased, but the ratio of protein nitrogen to total nitrogen remained fairly constant. A. A. ELDRIDGE.

Physical and chemical changes in celery during storage. L. W. CORBETT and H. C. THOMPSON (Proc. Soc. Amer. Hort. Sci., 1925, 346—353).—Calcium pectate and pectose are converted into pectic acid and pectin more rapidly in celery treated with nitrogenous fertilisers only than after treatment with phosphorus and potash. Changes in the soluble and insoluble nitrogen, and in reducing and total sugars, are recorded. A. A. ELDRIDGE.

Composition of loganberry juice. J. A. DAWSON (Canad. Chem. Met., 1926, 10, 81—84).—The relatively high acid and pectin contents of the loganberry make it a useful ingredient for jams and jellies, and it is probable that the fresh berry has a positive anti-scorbutic value. The juice amounts to about 70% by weight of

the berry, and the results of analyses of juice from various sources are given, approximate figures being as follows:—$d^{15 \cdot 6}$ 1·038—1·048; total solids, 9·9—12·6%; reducing sugars, 5·6—7·9%; sucrose, 0·01—0·08%; alcohol precipitate, 0·36—0·56%; citric acid, 2·09—2·30%; ash, 0·312—0·514%; phosphate as P_2O_5, 20·0—33·4 mg. per 100 c.c. C. O. Harvey.

Detection of saccharin in foods. L. Soep (Chem. Weekblad, 1926, **23**, 361—362).—Saccharin may be extracted from cakes, pastries etc., by crushing with slaked lime and extracting with alcohol; the extract is subjected to a lengthy process of purification, and saccharin, if present, is eventually obtained by ether extraction from an aqueous solution acidified with phosphoric acid. This final extract contains also any aromatic acids present or formed during the treatment, and is tested for saccharin by taste, the resorcinol test, hydrolysis, etc. S. I. Levy.

Detection of preservatives containing fluorine in foods. H. Lührig (Pharm. Zentr., 1926, **67**, 513—518, 531—535).—The methods of detecting the use of fluorine derivatives as preservatives in foodstuffs etc. are reviewed. The author advises the general use of tests depending on the method of Meyer and Schulz (Z. angew. Chem., 1925, **38**, 203—206), in which the presence of fluorides is shown, in the absence of silicates and phosphates, by the precipitate given by lanthanum acetate in the presence of acetic acid and ammonium acetate, the test being confirmed by the glass-etching method. Tests for fluorides in fats, butter, margarine, milk, beer etc., are worked out on this basis. B. Fullman.

Detection of colouring matters in foods. L. Soep (Chem. Weekblad, 1926, **23**, 362—365).—Metanil Yellow and Orange IV, the latter not being regarded in Holland as poisonous, are distinguished by means of cobalt acetate, manganous chloride, and silver nitrate. The material is shaken with cold 70% alcohol, the residue after filtration and evaporation dissolved in water, and the test solutions added to separate quantities. The colorations and precipitates are described. The results of numerous experiments to differentiate New Blue R and New Blue B from Water-soluble Induline are tabulated; the most suitable reagents are potassium iodide, potassium thiocyanate, and zinc iodide; the colour reactions with the common acids and alkalis on wool have been repeated, and modified results are given. S. I. Levy.

Loss of chlorine in the incineration of foodstuffs. J. D. Filippo and W. Adriani (Z. Unters. Lebensm., 1926, **51**, 374—375). J. Drost (*Ibid.*, 375—376).—Filippo and Adriani do not agree with a previous statement of Drost that loss of chlorine during incineration is due to mineral matter, especially acid phosphates, and state that there is considerable loss in phosphate-free material; also that chlorides when heated with ash-free carbon compounds, or with pure carbon itself, react and give an alkaline ash, due to sodium carbonate. Drost agrees with these statements and states that on heating a mixture of sodium chloride and potassium chloride with pure lactose there was a loss of chlorine of 22—33%. For the determination of chlorine in foodstuffs he recommends methods which do not entail incineration. W. G. Carey.

Effect of milk salts on the whipping ability of ice-cream mixes. H. H. Sommer and D. M. Young (Ind. Eng. Chem., 1926, **18**, 865—866).—Sodium citrate added in an amount greater than 0·4% to the ice cream before the ageing of the mix causes a marked increase in the whipping ability of the mix. Similar results are obtained by the addition of 0·7% of disodium phosphate; 0·5% of calcium lactate shows a marked lowering effect. These results indicate the possibility of effecting economies in ice-cream manufacture. B. W. Clarke.

"Finger and toe" disease of swedes. [Composition of swedes.] T. Whitehead (Welsh J. Agric., 1925, **1**, 176—184).—The roots of Magnum Bonum, Danish 25, Danish 4, and Yellow Turnip varieties of swedes yielded results within the following limits:—dry matter, 8·5—11·1; total sugar, 3·32—6·25; reducing sugar in juice, 3·30—6·20; sucrose in juice, 0·15—0·31%. A. A. Eldridge.

Acid formation in silage. K. Schmidt (Landw. Jahrb., 1926, **63**, 776—779; Chem. Zentr., 1926, II., 837; cf. following abstract).—The conclusion of Mangold is confirmed that the production of acids in silage reaches a maximum after two days. Lucerne silage made in evacuated silos or in an atmosphere of an inert gas contains considerably less acetic and butyric acids than under normal conditions, and the breaking down of nitrogen compounds to ammonia is diminished, *i.e.*, the silage is of better feeding value. Addition of very dry material, *e.g.*, maize straw, to the green fodder similarly reduces the formation of butyric acid. C. T. Gimingham.

Preservation of young green fodder. II. K. Schmidt (Leopoldina, 1926, **2**, 107—116; cf. preceding abstract).—Laboratory and large-scale experiments were made on the effect on the composition of lucerne silage of evacuating the silos after filling and then introducing carbon dioxide or carbon disulphide vapour. Feeding experiments with sheep and cows showed that the silage made in this way gave satisfactory results. In the laboratory experiments, the formation of butyric acid was entirely suppressed. Treatment with carbon disulphide also reduced the amounts of acetic acid and ammonia produced. Samples of the gases in the silos were analysed at intervals. There was a markedly greater production of hydrogen in the silage treated with carbon dioxide than in that treated with carbon disulphide; and, in the large silo, an increased amount of hydrogen in the lowest layer. Temperature changes and the rate of formation of acids in the silage are also discussed. C. T. Gimingham.

See also A., Sept., 959, **Tryptophan–aldehyde reaction. Determination of tryptophan in proteins** (Komm). 970, **Liberation of phosphorus from caseinogen by enzymes and other agents** (Rimington and Kay); **Effect of heat on milk** (Magee and Harvey). 980, **Fearon's "pyrogallol" test as possible basis for determination of vitamin-*A*** (Willimot and Moore); **Solubility of vitamin-*B* in benzene**

(WILLIAMS and WATERMAN). 981, **Antirachitic substances. Polymerisation of cholesterol** (BILLS and McDONALD); **Dietary requirements for reproduction** (SURE). 982, **Lecithin, kephalin, and so-called cuorin of soya bean** (LEVENE and ROLF).

Determination of fat in oil-cake. ZANDER.—See XII.

Examination of milk sweets. FINCKE.—See XII.

PATENTS.

Manufacture of food [and other] products from fish. SOC. FRANC. DES PROD. ALIMENTAIRES AZOTÉS (E.P. 246,148, 13.1.26. Conv., 14.1.25).—The fish is autolysed at 37—55° in presence of 5—10% of sodium chloride, the temperature being lower the greater the proportion of salt used. A portion of the oil collects at the surface and is recovered, *e.g.*, by decantation. The residue is sieved and from the skeleton débris are obtained more oil and a powder constituting a manure rich in nitrogen and phosphorus. The liquid portion is filtered, and the filter residue is used for tanning or tawing or as a further source of oil and manure. The filtrate may constitute the final food product or may be concentrated to a paste. H. HOLMES.

Working up slaughterhouse offal, animal carcases, and fish. W. STEINMANN (E.P. 256,162, 20.4.26). —The material is sterilised and disintegrated by means of steam, and the resulting pulp is dried, and then immediately extracted with a fat solvent, such as carbon tetrachloride or benzine. A better separation of the fat is obtained than with other steam processes, and there is no loss of protein. C. O. HARVEY.

Bulgarian milk. H. BUEL (U.S.P. 1,593,899, 27.7.26. Appl., 30.4.20).—Milk is freed from cream and inoculated with *Bac. lacticus Bulgaricus*, together with a type of *paralacticus*, *Streptococci lacticus*, or *Diplococci lacticus* and kept at 30—36°. The cream after inoculation as above, but without the *Bacillus lacticus Bulgaricus*, is mixed with the cultured milk, cooled to 13—18°, and, finally, to 2—8°, at which temperature the product is held for a time. C. RANKEN.

Manufacture of leavened bread. H. A. KOHMAN and R. IRVIN, Assrs. to FLEISCHMANN Co. (U.S.P. 1,593,977, 27.7.26. Appl., 13.12.19).—A substance used in the manufacture of leavened bread is prepared by cooking hominy in an aqueous solution of ammonium sulphate until the solution is absorbed by the hominy, adding calcium carbonate, and inoculating with an *Aspergillus* fungus, which is grown until the hominy is bound together. The product, after being dried at a temperature which does not injure the enzymes, is ground. C. RANKEN.

Manufacture of soluble cocoa. C. and G. BREHIER and F. P. JEAN (F.P. 601,199, 24.10.24).—Cocoa powder is treated with proteolytic enzymes to render soluble the contained proteins. C. RANKEN.

Pectin preparation. A. E. A. P. LIOT and L. A. MACÉ (F.P. 604,529, 8.1.25).—A liquid obtained by extraction of apple and pear pulp in presence of an alkaline-earth carbonate is caused to coagulate by treatment with an extract containing pectase obtained from plants (lucerne etc.).

Purification of flour. L. G. PATROUILLEAU (F.P. 604,950, 22.10.25).—The flour is purified by an oxidising agent (NO_2) which converts the allyl sulphide in the husks into allyl sulphoxide, an odourless liquid product, soluble in water. C. RANKEN.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Suitability of chloroform for alkaloidal assay. H. R. WATKINS and S. PALKIN (Ind. Eng. Chem., 1926, **18**, 867—869).—Purity tests commonly used to determine the suitability of chloroform for alkaloidal assays are shown to be inadequate, the specimens giving the best results by these tests often giving the largest errors. The suitability of a given sample is best determined on actual test on the alkaloid by the automatic, continuous extraction method. Unsuitable samples may be improved by prolonged refluxing with brucine, followed by recovery of the chloroform from the extract by distillation. In all cases extraction by the continuous process using benzene as a solvent gave excellent results, and this solvent is recommended as a substitute for chloroform in alkaloidal assays. J. W. BAKER.

Detection and approximate evaluation of ergot. A. TSCHIRCH (Pharm. Acta Helv., 1926, **1**, 89—90; Chem. Zentr., 1926, II., 927).—If a solution of ferric chloride in concentrated sulphuric acid be introduced under a glacial acetic acid solution of histamine or tyramine (both commercially known as tenosin) an orange-yellow ring is formed; a glacial acetic solution of a trace of ergosterol shows a green fluorescence if a layer of strong sulphuric acid be introduced under it. For the identification and approximate evaluation of ergot, it is treated with ether and ammonia. A red solution is formed (sclererythrin). After 2 hrs. the ether is separated and distilled, the solution of the residue in glacial acetic acid being treated as above with a solution of ferric chloride in strong sulphuric acid. A bluish-violet coloration (ergotamine) should appear at the zone of contact, but hardly any or no yellow colour (amines derived from proteins), and the acetic acid layer should show a green fluorescence (ergosterol). For this test the ergot should contain at least 0·2% of ergotamine. B. FULLMAN.

Suitability of monel metal for vanilla flavouring containers. T. E. HOLLINGSHEAD and T. J. OTTERBACHER (Ind. Eng. Chem., 1926, **18**, 871—872).—When various types of vanilla flavours, prepared from Bourbon beans and, in some cases, fortified with vanillin and coumarin, were placed in contact with monel metal, the extracts showed a slightly weaker flavour at the end of 6 weeks, although the odour seemed the same. At the end of 10 weeks both odour and flavour were weaker. Throughout 12 weeks no precipitate was formed, and the colour remained unchanged. The action in the light was the same as in the dark. The polished surface

of the metal was dulled at the end of the eighth week. The standard extract produced a greater effect than did the fortified flavours, while the corrosion was practically twice as great in the light as in the dark. The normal lead number of the true vanilla bean extracts was higher after treatment than before. This is probably due to the resins forming compounds with nickel and copper, which precipitate more lead. In the fortified extracts the normal lead number fell considerably. C. Ranken.

Pharmaceutical lactic ferment preparations. J. Placeres (Rev. Fac. Cien. Quím., 1926, 4, 73—93).—Certain commercial lactic ferment preparations are either inactive, or else harmful on account of their proteolytic properties. The method proposed for determining the activity of lactic ferment preparations consists in incubating 100 c.c. of skim milk containing 3% of lactose with 0·5 g. of the preparation at 37° for 48 hrs., and determining the acidity by titration with standard sodium hydroxide, using phenolphthalein as indicator. A control determination is also made without the preparation. Butyric acid is produced by the decomposition of fats present in the medium, and is not a secondary product of lactic fermentation. Commercial lactic acid preparations do not yield harmful volatile products. G. W. Robinson.

Aliphatic tertiary alcohols and their industrial possibilities. H. S. Davis and W. J. Murray (Ind. Eng. Chem., 1926, 18, 844—846).—The physical and chemical properties of the tertiary aliphatic alcohols are compared with those of the primary and secondary, and their possible industrial uses outlined. As a general rule the b.p., density, and solubility in water of the tertiary alcohol are similar to those of the homologous secondary alcohol with one carbon atom less, or the primary alcohol with two carbon atoms less. Chemically, the tertiary alcohols are characterised by their ease of dehydration (*e.g.*, *tert.*-butyl alcohol yields *iso*butylene on treatment with oxalic acid), lability of the hydroxyl group, resistance to oxidation, stability towards halogens, and very slow reaction with sodium. With acetyl chloride *tert.*-butyl alcohol yields *tert.*-butyl chloride and acetic acid. J. W. Baker.

Comparative tests on the sensitiveness of some usual reagents for methyl alcohol. B. Olszewski (Rocz. Farm., 1925, 3, 77—88; Chem. Zentr., 1926, II., 801—802).—The author has investigated dilute solutions of methyl alcohol in water and ethyl alcohol (the latter sometimes containing fusel oil) by oxidising with potassium permanganate and testing for formaldehyde. The most characteristic test is that with apomorphine hydrochloride in sulphuric acid, which will indicate 1% of methyl alcohol in water and ethyl alcohol mixtures after direct oxidation, 0·2% on oxidation after one distillation, and 0·1% on oxidation after two distillations. In the absence of other aldehydes, 0·001% of formaldehyde (or 0·02% of methyl alcohol on oxidation) gives a positive reaction on pouring the liquid on to the apomorphine solution without mixing, a coloured ring being formed. B. Fullman.

Acidimetric and thiocyanometric determination of mercuric chloride [in sublimate pastilles]. E. Rupp with K. Müller and P. Maisz (Pharm. Zentr., 1926, 67, 529—531).—Mercuric chloride in pastilles containing it may be thus determined: the solution of the substance is added to alkaline (sodium hydroxide) hydrogen peroxide, whereby the mercuric chloride is reduced to mercury thus: $HgCl_2+2NaOH=HgO+2NaCl+H_2O$, followed by $HgO+H_2O_2=Hg+H_2O+O_2$. The excess of sodium hydroxide is titrated with hydrochloric acid. Alternatively, the mercury obtained as above is coagulated by addition of hydrochloric acid, the supernatant liquid decanted, and the mercury dissolved in concentrated nitric acid, excess of the latter being removed by addition of 1% permanganate solution to permanent coloration. After decolorising with a crystal of ferrous sulphate, iron alum solution and dilute nitric acid are added, and the solution is titrated with standard ammonium thiocyanate. Organic substances etc. retard the coagulation of the mercury, and in the case of pastilles the acidimetric method is therefore preferable. B. Fullman.

Loss of free iodine from alcoholic solutions of varying purity and the effect of potassium iodide. R. M. Carter (Ind. Eng. Chem., 1926, 18, 827—828).—The loss of iodine from ethyl alcoholic solutions on keeping is greatly influenced by the purity of the solvent, being much greater when low-grade alcohol is used. Except with such alcohol, addition of 75% of the theoretical amount of potassium iodide required to form KI_3 largely inhibits the loss, which is then less than 2% after 1 year. The loss of iodine from solutions in commercial methyl alcohol is due mainly to the impurities present in the solvent. J. W. Baker.

Geraniol and its determination; notes on citronellol. L. G. Radcliffe and E. Chadderton (Perf. Ess. Oil Rec., 1926, 17, 254—264, 350—355).—Methods for the determination and purification of geraniol and citronellol have been examined and are critically reviewed. For the determination of geraniol in commercial samples, the usual acetylation method is the best. The method of Verley and Bölsing (A., 1902, ii., 54), *i.e.*, acetylation in presence of pyridine, is shorter, but gives results about 2% low. A new method is described in which phthalic anhydride in presence of pyridine is used for acylation. This gives results about 2% low, but is more convenient than the pyridine–acetic anhydride method. For the determination of citronellol in commercial samples the acetylation method is again superior, but the new method gives results almost as good. In presence of pyridine, phthalic anhydride is selective in its action on alcohols, linalool, for example, not being attacked, and this method is the best for the determination of free geraniol in citronella oil, but for the total alcohols the figure given by Verley and Bölsing's method is the most accurate, Schimmel's method often giving variable results. For the determination of citronellol in presence of geraniol, the formylation process is unsatisfactory, and it is suggested that the process of Tiemann and Schmidt (A., 1896, i, 382), *i.e.*, isolation of the citronellol with phosphorus trichloride, would give a more correct figure. A rapid test for determining the

purity of geraniol samples has been devised, based on the speed of the reaction of geraniol with finely ground calcium chloride and the hardness of the product; the presence of quite small amounts of impurities has a very pronounced effect on the rate of formation and consistency of the additive product, which has been shown to have the constitution, $CaCl_2,2C_{10}H_{17}\cdot OH$. E. H. SHARPLES.

Effect of the hydrogen-ion concentration of the soil on the formation and composition of essential oil of wood sage. H. and H. DEEL (Bull. Soc. chim., 1926, [iv], **39**, 946—948).—Under comparable conditions, the largest crop of wood sage, *Salvia sclarea*, is obtained from a soil of p_H approx. 4·5, and the resulting essential oil has also a maximum ester content. The content of essential oil in the plants is, however, almost independent of the p_H variation. Plants from a soil of p_H 4·65 yielded 0·157% of essential oil, d^{15} 0·897, $[\alpha]$ — 18·4°, n 1·4757, acid value 0·28, containing 46·55% of esters. H. E. F. NOTTON.

Determination of essential oils in spices. C. GRIEBEL (Z. Unters. Lebensm., 1926, **51**, 321—324).—A mixture of 10 g. of ground spice with 300 c.c. of distilled water is distilled, and 200 c.c. of the condensed distillate are received in a separating funnel. This distillate is then shaken with 60 g. of common salt and 20 c.c. of pentane, and after complete settling, the pentane layer is transferred to a weighed flask, and the pentane is evaporated on a water bath, the last traces being removed by a dry air blast. The residue of essential oil is dried in a desiccator and weighed. The pentane used must not leave a residue on evaporation, and must be free from non-volatile or high-boiling impurities. W. G. CAREY.

German essential flower-extract oils. W. TREFF, F. RITTER, and H. WITTRISCH (J. pr. Chem., 1926, [ii], **113**, 355—360).—Essential oils from a number of German-grown plants, obtained by extraction with light petroleum, solution in alcohol, and triple steam distillation (cf. Soden, A., 1904, i, 515), have been examined. Leaves of *Viola rossica*, "Queen Charlotte," yield 0·0166% of yellowish oil with a stupefying odour, optically inactive, d^{15} 0·912, acid value 52, ester value 76·1, acetyl value 172. Flowers, with calyx-leaves, of the carnation, *Dianthus caryophyllus*, L., yield 0·0498% of a pale yellow oil with a pleasant carnation odour, d^{15} 1·010, α — 0° 36′, acid value 28, ester value 132, acetyl value 249. Flowers of the German jasmin, chiefly *Coronarius*, yield 0·06% of a yellowish oil, with a powerful, characteristic odour, differing from that of true jasmin oil, optically inactive, d^{15} 0·947, acid value 28, ester value 73, acetyl value 224, probably containing methyl anthranilate (cf. Verschaffelt, Chem. Weekblad, 1908, 25). Yellow lupin flowers, *Lupinus luteus*, L., yield 0·0195% of a yellowish oil with a penetrating, sweetish odour, d^{15} 0·900, α_D + 7° 30′, acid value 38, ester value 31, acetyl value 143 (cf. Schimmel's Ber., 1926, 195). Flowers of broom, *Genista tinctoria*, L., yield 0·0364% of a yellowish oil, of oppressive odour, d^{15} 0·9335, α_D — 9° 10′, acid value 18, ester value 35, acetyl value 156. F. G. WILLSON.

Evolution of synthetic medicinal chemicals. H. V. ARNY (Ind. Eng. Chem., 1926, **18**, 949—952).

Fifty years' progress in standardisation and evaluation of medicinals. P. N. LEECH (Ind. Eng. Chem., 1926, **18**, 953—954).

Development of the aromatic chemical industry from 1876 to 1926. M. SZAMATOLSKI (Ind. Eng. Chem., 1926, **18**, 933).

See also A., Sept., 954, **Sesquiterpene family in camphor blue oil** (KOIKE). 959, **Synthesis of 5-β-aminoethylacridine** (JENSEN and HOWLAND). 963, **Alipine hydrochloride** (SEVILLA); **Alkaloids of *Calumba* root** (SPÄTH and BURGER). 964, **Synthesis of cryptopine and protopine** (HAWORTH and PERKIN). 965, **Alkaloids of *Corydalis cava*** (SPÄTH and MOSETTIG); **Colour reactions of morphine** (EKKERT); **Reactions of certain nitro-compounds with alkaloids** (NAVARRO); **Synthesis of iodine compounds of the salvarsan group** (MACALLUM). 967, **Determination of small quantities of water in methyl alcohol** (RISING and HICKS); **Thalleioquinine reaction** (HARGREAVES); **Electrometric titration of alkaloids. Application of the quinhydrone electrode** (WAGENER and MCGILL). 974, **Chemical constitution and physiological action. Position isomerism in relation to miotic activity of some synthetic urethanes** (STEDMAN). 979, **Preparation and properties of purified diphtheria toxoid** (WATSON and LANGSTAFF). 980, **Preparation, properties, and source of parathyroid hormone** (DAVIES, DICKENS, and DODDS). 981, **New Zealand pepper-plant** (FINLAY). 982, **Kombe-strophanthin-*β* and other Kombe-strophanthins** (JACOBS and HOFFMANN); **Composition of Scilla and its tonicardiac principle** (HENRIJEAN and KOPACZEVSKI).

Additive products of iodine monobromide and hypoiodous acid with unsaturated compounds. HOLDE and GORGAS.—See XII.

PATENTS.

Manufacture of benzimidazoles. FARBENFABR. VORM. F. BAYER & CO. (E.P. 243,766, 1.12.25. Conv., 1.12.24.).—*o*-Phenylenediamine or its homologues and derivatives react with alkoxy-acids or their derivatives, yielding, directly or via an intermediate compound, 2-alkoxymethylbenzimidazoles, the latter being antipyretics. For example, 1 pt. of *o*-phenylenediamine and 3 pts. of ethoxyacetic anhydride are heated together for 5 hrs. at 120°. Addition of water precipitates the diacyl-*o*-phenylenediamine, which on heating for 1 hr. at 180° yields *2-ethoxymethylbenzimidazole*, m.p. 153°. The following are also described :—*2-ethoxymethyl-5-ethoxybenzimidazole hydrochloride*, m.p. 162°; *2-phenoxymethylbenzimidazole*, m.p. 190°, and *2-phenoxymethyl-5-ethoxybenzimidazole*, m.p. 129°. B. FULLMAN.

Manufacture of alkylated aromatic sulphonic acids. I. G. FARBENIND. A.-G. (E.P. 246,817, 9.1.26. Conv., 30.1.25.).—One or more n-butyl groups may be introduced into the nuclei of aromatic sulphonic acids by condensing the latter with n-butyl alcohol, with or without condensing agents, such as sulphuric acid, or

by using the salts of the sulphonic acids, in presence of a condensing agent, or by condensing aromatic hydrocarbons with *n*-butyl alcohol in the presence of a condensing agent, subsequently (or when sulphuric acid is used, simultaneously) sulphonating. For example, naphthalene, *n*-butyl alcohol, and sulphuric acid of *d* 1·84 are heated together. From the reaction product butylated naphthalenesulphonic acid may be isolated.

B. FULLMAN.

Manufacture of the pure glucoside of *Bulbus scillæ*. A. HOME-MORTON. From F. HOFFMANN-LA ROCHE & Co. (E.P. 255,689, 14.10.25).—The pure glucoside of *Bulbus scillæ* (sea-onions) is obtained by removing the glucoside from the aqueous extract of the fresh or dried drug by means of an adsorbing agent such as charcoal or fuller's earth, and extracting the latter with an anhydrous organic solvent. For example, the charcoal is extracted with hot chloroform, the latter distilled off, the residue dissolved in methyl alcohol, and the inactive substances present removed by extraction with light petroleum. The methyl alcohol is distilled off and the residue dissolved in absolute alcohol, from which the pure glucoside is precipitated by pouring into ether.

B. FULLMAN.

Process for making arsonophenylaminoalcohols. E. C. R. MARKS. From ABBOTT LABORATORIES (E.P. 255,971, 6.5.25).—ω-Chloroalkyl chloroformates condense with equimolar proportions of arsanilic acids or their substituted derivatives to give ω-chloroalkyl arsinophenylcarbamates, which, on refluxing with excess of alkali and acidification yield arsinophenylaminoalcohols of the type $H_2O_3As \cdot R \cdot NH \cdot [CH_2]_n \cdot OH$, and where R is a benzene (or substituted benzene) nucleus. These substances are used in the treatment of syphilis, especially in the tertiary stage. The ω-chloroalkyl chloroformates are prepared from alkylene chlorohydrins of the type $Cl \cdot [CR_2]_n \cdot OH$ (where R = H or alkyl) and phosgene. For example, 21·7 g. of *p*-arsanilic acid are dissolved in 200 c.c. of water containing 4 g. of sodium hydroxide, and 14·2 g. of β-chloroethyl chloroformate are added, the temperature being kept below 35°. After acidification, the precipitated β-*chloroethyl* p-*arsinophenylcarbamate* is filtered off. On refluxing it for 4 hrs. with 50 c.c. of 20% sodium hydroxide and 50 c.c. of water, cooling, and adding hydrochloric acid, β-[p-*arsinophenylamino*]*ethyl alcohol*, m.p. 171—173° (decomp., corr.), separates. The following are also described :—γ-[p-*arsinophenylamino*]*propyl alcohol*, m.p. 167—168° (decomp., corr.), from γ-*chloropropyl* o-*arsinophenylcarbamate*, m.p. 239—240° (decomp., corr.); β-[o-*arsinophenylamino*]*ethyl alcohol*, m.p. 145—146° (decomp., corr.), from β-*chloroethyl* o-*arsinophenylcarbamate*, m.p. 156—157° (decomp., corr.); γ-[o-*arsinophenylamino*]*propyl alcohol*, m.p. 84—85° (decomp., corr.), from γ-*chloropropyl* o-*arsinophenylcarbamate*, m.p. 139—140° (decomp., corr.); and β-[2-*methyl*-5-*arsinophenylamino*]*ethyl alcohol*, m.p. 144—145° (decomp., corr.), from β-*chloroethyl* 2-*methyl*-5-*arsinophenylcarbamate*, m.p. 194—195° (decomp., corr.). B. FULLMAN.

Sulphonated thianthrens useful as pharmaceutical compounds. H. HERMINGHAUS, Assr. to WINTHROP CHEMICAL Co. (U.S.P. 1,589,390, 22.6.26. Appl., 24.5.24. Conv., 4.8.23).—Thianthren, or its derivatives, *e.g.* dimethylthianthren or the compounds obtained by the methods described in U.S.P. 1,426,430 and 1,427,182 (B., 1922, 786, 837), are treated with sulphuric acid or chlorosulphonic acid at 100—160° until the product is completely soluble in water. The sodium salt of the monosulphonated thianthren derivative thus obtained is readily soluble in water, and has a strong "antiphlogistical" action. T. S. WHEELER.

Catalytic oxidation of organic compounds. C. R. DOWNS (U.S.P. 1,589,632, 22.6.26. Appl., 21.1.24.).—In the oxidation of toluene to benzaldehyde by passing it in the vapour phase with air over an oxide of molybdenum at 525°, this optimum temperature is readily maintained and excess heat of reaction removed by surrounding the catalyst chamber with sulphur boiling under reflux at the pressure corresponding to that temperature. The method is of wide application in the temperature control of catalytic reactions. T. S. WHEELER.

Anæsthetic compound. R. ADAMS, E.E. DREGER, and E. H. VOLWILER, Assrs. to ABBOTT LABORATORIES (U.S.P. 1,590,792, 29.6.26. Appl., 22.8.24).—The *p*-aminobenzoates of amino-alcohols having the general formula $CHMe(NR_2) \cdot CH_2 \cdot OH$ (R=alkyl) have anæsthetic properties both in the form of the free base and as the mineral acid salt. They are prepared by treating the amino-alcohol in benzene solution with *p*-nitrobenzoyl chloride and reducing the nitro-group in the product. β-*Diethylamino*-n-*propyl alcohol*, b.p. 166—169°, d^{27} 0·8665, n_D^{24} 1·4305, β-*diethylamino*-n-*propyl* p-*nitrobenzoate hydrochloride*, m.p. 155—156°, β-*diethylamino*-n-*propyl* p-*aminobenzoate hydrochloride*, m.p. 159—160°, β-*di*-n-*butylamino*-n-*propyl alcohol*, b.p. 112—114/10 mm., d^{20} 0·8533, n_D^{18} 1·4426, β-*di*-n-*butylamino*-n-*propyl* p-*aminobenzoate monohydrochloride*, m.p. 200—201°, β-*diallylamino*-n-*propyl alcohol*, b.p. 145—147/123 mm., d^{20} 0·9103, n_D^{20} 1·4466, β-*diallylamino*-n-*propyl* p-*aminobenzoate monohydrochloride*, m.p. 182—183°; β-n-*butylallylamino*-n-*propyl alcohol*, b.p. 123—125/130 mm., and β-n-*butylallylamino*-n-*propyl* p-*aminobenzoate monohydrochloride* are described.

T. S. WHEELER.

Manufacturing benzoic acid from benzotrichloride. F. E. STOCKELBACH, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,591,245, 6.7.26. Appl., 26.5.21. Renewed 20.2.25).—700 pts. of calcium carbonate or other alkaline-earth carbonate are suspended in 3000 pts. of water at 80—90°, and 760 pts. of crude benzotrichloride are added with agitation during 3—4 hrs. Heating is continued for 1 hr. and the calcium benzoate formed is filtered off. T. S. WHEELER.

Preparation of micro-organisms free from nucleic acids, containing only lipoids and lipoproteins. BEHRINGSWERKE A.-G. (G. P. 424,657, 2.11.24).—The cells of micro-organisms are treated with mineral acids, to hydrolyse the nucleoproteins and all the acid proteins, the resulting acid-soluble products being washed out of the cells. The micro-organisms thus treated serve as intermediate products for therapeutic preparations. For example, pressed yeast is shaken for several hours in 6·5% nitric acid, and the simpler

nucleic acids are subsequently washed out with water. The cells then contain only lipoids and lipoproteins, and the yeast is free from purine bases. If pressed yeast be treated with 5% sulphuric acid or dilute hydrochloric acid the nuclear proteins remain in the cells.

B. FULLMAN.

Preparation of metal-containing yeasts and other micro-organisms. BEHRINGSWERKE A.-G. (G.P. 424,658, 4.11.24).—Micro-organisms are treated with solutions of metal salts, and the cells washed with distilled water till the wash-water is free from metal. The products are free from nucleic acids and are better tolerated therapeutically than the known metal nucleates or yeast–metal compounds. For example, a uniform suspension of yeast is poured into 2% silver nitrate, and after 24 hrs. the supernatant liquid decanted off and the silver–yeast washed with distilled water. It has a high silver content. If it be washed with physiological sodium chloride solution or tap water, almost all the silver is washed out from the cells, and simultaneously proteins pass out. Mercury-containing yeast cells are obtained if 2% mercuric chloride be used. B. FULLMAN.

Preparation of aqueous emulsions and sols. E. MERCK, Assees. of W. EICHHOLZ (G.P. 424,748, 31.3.22. Addn. to 330,673, B., 1924, 35).—In place of the camphor of the original patent, substances insoluble in water but soluble in lipoids, fats, or lipoid solvents or in lipoid solutions (except cholesterol) are used to emulsify fatty acid salts of bismuth, copper, silver, mercury, quinine and its derivatives. For example, bismuth linoleate and lecithin are dissolved in ether, the ether is evaporated off, and the residue emulsified with distilled water.

A. DAVIDSON.

Preparation of aqueous emulsions and sols. E. MERCK, Assees. of W. EICHHOLZ (G.P. 426,743, 31.3.22. Addn. to 424,748; cf. preceding abstract).—The lipoids, or the lipoids and fatty acid salts together, specified in the previous patent may be emulsified by dissolving them in easily volatile solvents which are miscible with water, adding water to the solution, and driving off the solvent. For example, lecithin is dissolved in warm alcohol, the solution filtered, poured into distilled water, and evaporated in a vacuum. There is thus obtained a 10% lecithin emulsion completely free from alcohol, of high dispersion and good stability.

A. DAVIDSON.

Preparation of 5:7-di-iodoindole-3-acetic acid and its homologues. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of W. SCHOELLER and K. SCHMIDT (G.P. 425,041, 28.5.24).—2:4-Di-iodophenylhydrazine is condensed with aldehydo-acids or their esters and the condensation products are heated with alcoholic acids. For example, γ-aldehydopropionic acid is refluxed at 100° with an acetic acid solution of 2:4-di-iodophenylhydrazine for 2 hrs. The resulting 2:4-*di-iodophenylhydrazide of the γ-aldehydopropionic*-2:4-*di-iodophenylhydrazone*, m.p. 185°, is boiled with alcoholic sulphuric acid for 6 hrs. Di-iodophenylhydrazine splits off and 5:7-*di-iodoindole*-3-*acetic acid*, m.p. 106°, is formed. Similarly, from δ-aldehydobutyric acid is obtained 5:7-*di-iodoindole*-3-*propionic acid*, m.p. 111°. The products find therapeutic application. A. DAVIDSON.

Preparation of substances which alone or with other greasy substances form salves. A. ENGEL (G.P. 425,331, 20.6.23).—The fatty acids from marine animal fats are treated with halogens in the presence of red phosphorus or the halides or oxyhalides of phosphorus or sulphur, the halogenated derivatives obtained being heated with water or ammonia. The products swell up in water, in which they are insoluble. Vaseline or animal fat mixed with 5% of these products takes up large quantities of water, forming a stable grease. For example, the dry fatty acids from fish oil are ground with red phosphorus. Chloroform, benzene, or light petroleum is added, and chlorine passed in. On pouring into water, a mass is obtained which melts to a grease on heating to 100°; or the chlorinated liquid may be poured into aqueous ammonia and boiled; or gaseous ammonia may be passed into the chlorinated liquid. B. FULLMAN.

Manufacture of technical triacetin. M. SONN (G.P. 425,611, 30.4.24).—A mixture of glycerol, fused sodium acetate, and glacial acetic acid is treated at 100—110° with a slow current of anhydrous hydrogen chloride; the liquid is separated from the solid sodium chloride by suction, and the crystals are washed with glacial acetic acid. The triacetin is recovered from the filtrate by distillation without steam at atmospheric pressure. By redistillation a yield of 85—95% of the theoretical of colourless and odourless triacetin is obtained free from chlorine and acrolein, whilst the excess of acetic acid is recovered in the glacial form for use again in the process. A. R. POWELL.

Preparation of 6-nitro-3:4-dimethoxytoluene (6-nitrohomoveratrole). E. MERCK, Assees. of M. OBERLIN (G.P. 425,739, 7.8.24).—6-Nitro-3:4-dimethoxybenzaldehyde is condensed with hippuric acid and the oxazolone derivative so formed is hydrolysed with alkali. For example, a mixture of 6-nitro-3:4-dimethoxybenzaldehyde, hippuric acid, anhydrous sodium acetate, and acetic anhydride is gently boiled under reflux for 1 hr. The cooled mixture is extracted with boiling water or acetone and crystallised from ethyl acetate and then from alcohol, when 2-*phenyl*-4-[3′:4′-*dimethoxy*-6′-*nitrobenzylidene*]*oxazolone*, m.p. 220·5—221°, is obtained. The crude oxazolone derivative is boiled under reflux with 10% sodium hydroxide for 4 hrs. to obtain 6-*nitro*-3:4-*dimethoxytoluene*, m.p. 117·5—118°; yield 85%. It is used as intermediate in the synthesis of therapeutic compounds. A. DAVIDSON.

Preparation of carbamide from cyanamide. H. GOLDSCHMIDT & VON VIETINGHOFF CHEM.-TECHN. GES.M.B.H. (G.P. 426,671, 9.3.20).—Adsorption compounds of the heavy metals with the hydrogels of silicic acid, zirconium dioxide, titanic acid, thorium dioxide, and the like are used as catalysts. They are prepared by filtering an alkaline solution of the heavy metal concerned through the gel. For example, a 25% cyanamide solution is mixed with copper–silicic acid, obtained by filtering an ammoniacal cupric oxide solution through silicic acid gel, and kept for some time with frequent agitation. After 3 hrs., 39%, after 24 hrs. 54%, and after 72 hrs. over 70% of the cyanamide is converted into carbamide. A. DAVIDSON.

Preparation of tartaric acid. DIAMALT-A.-G. (G.P. 426,864, 6.10.20, and 427,415, 26.11.21).—Dextrose in aqueous solution or suspension is treated with hydrogen peroxide or other peroxides, *e.g.*, those of potassium, sodium, calcium, or barium, in presence of organised catalysts, especially peroxydases and oxydases. As peroxydase solution, an aqueous extract of dormant corn embryos, especially rye embryos, is used. The process is carried out in presence of carbonates of the alkaline-earths, and pure carbon dioxide is passed through the reaction mixture during oxidation. For example, to a 5—10% dextrose solution at 25—30° a concentrated fresh peroxydase solution is added and, slowly and regularly, an aqueous solution of pure hydrogen peroxide run in, with constant stirring. Calcium carbonate is added to neutralise the resulting acid which, at a certain p_H, would stop the action of the catalyst. Finally, the liquid is heated to boiling point, when calcium tartrate separates. The yield of calcium tartrate is 40% of the dextrose used, but this is raised to 45% if pure carbon dioxide is passed in during oxidation. A. DAVIDSON.

Preparation of citric and other organic acids [by fermentation]. R. FALCK (G.P. 426,926, 31.5.21).—*Hyphomycetes* (thread fungi) are allowed to grow on solid substrates containing starch, and the mixture is worked up in the usual way for the resulting organic acids. Since *hyphomycetes* of the species *Aspergillus, Citromyces,* and *Penicillium* are not sensitive to tannins and poisons, starch-containing materials unsitable for food or fodder purposes, such as horse-chestnuts etc. may be used. Examples are given of the preparation of a mixture containing citric, succinic, malic, and tartaric acids from wheat flour, which is rubbed up with ammonium nitrate, calcium carbonate, and water, spread out hot on glass plates, and, after cooling, inoculated with *Citromyces D.* After keeping for 11 days at 13°, the mass is dried and powdered. The yield of acid is 25%. (Cf. Falck and van Beyma thoe Kingma, B., 1924, 649.)
A. DAVIDSON.

Preparation of quinine solutions suitable for subcutaneous injection. CHEM.-PHARM. A.-G. BAD HOMBURG (G.P. 427,668, 15.7.24).—Stable quinine solutions of high concentration in olive oil and similar oils may be obtained by the addition of terpenes or of essential oils containing them. For example, a 10% solution of quinine may be obtained by dissolving quinine in terpineol and adding olive oil. B. FULLMAN.

Method for working up animal organs. G. EICHELBAUM (G.P. 428,531, 1.12.22).—Animal organs are mixed with efflorescent indifferent salts, and the plasmolysing mixtures are dried by warming them in a stream of dry, warm air, with or without a vacuum. Stable preparations are obtained, the active components being unchanged. Extraction with solvents yields the lipoids, *e.g.*, the phosphatides dissolved in fat may be thus obtained, and on addition of indifferent fat and removal of the solvent may be used for injection. The hormones are extracted from the dry, fat-free residue with water, albumin being removed by concentration and excess salts by intense cooling. During the working up small quantities of phenol may be added. For example, finely ground testicle is mixed with anhydrous sodium sulphate, and dried at 40—50° as above. After a few hours the material is dry, friable, and almost indefinitely stable. B. FULLMAN.

Antiseptic anæsthetic medicament. F. K. THAYER, Assr. to ABBOTT LABORATORIES (U.S.P. 1,596,259, 17.8.26. Appl., 27.2.24).—See E.P. 230,089; B., 1925, 900.

Manufacture of methyl alcohol (F.P. 605,530).—See II.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitivity; a colloid-chemical problem. S. E. SHEPPARD (Colloid Symposium Monograph, 1925, **3**, 76—98; Chem. Abstr., 1926, **20**, 1959).—The accessory sensitivity of high-speed silver bromide in the blue and violet is not due to an optical sensitiser but follows the same absorption as that of the silver halide; hence the sensitivity-promoting substance does not affect the photochemical reaction. Evidence is adduced that this substance consists of silver sulphide nuclei acting by earlier formation of development nuclei in the production of the latent image.
A. A. ELDRIDGE.

Effect of desensitisers in development. J. I. CRABTREE and M. L. DUNDON (Brit. J. Phot., 1926, **73**, 404—406, 418—421, 434—437).—The efficiency of certain Wratten safelights for use with different types of emulsion is discussed. The essential requirements of practical desensitisers are considered, and the properties of Phenosafranine, Pinakryptol Green, Pinakryptol Yellow, Basic Scarlet N, and Aurantia are studied in the light of these requirements. The limits of safety in the use of Pinakryptol Green with motion-picture negative and panchromatic emulsions are determined. Desensitising by Pinakryptol Green is directly proportional to the concentration of dye over the ranges studied. Desensitising with Pinakryptol Green reduces the colour sensitivity of a sensitised emulsion much more than it does the blue sensitivity. Pinakryptol Yellow is more effective and Phenosafranine less effective in lowering colour sensitivity. With increased time of bathing in desensitiser, sensitivity falls off very rapidly at first, but after about 5 min. the rate of decrease is very low. The comparative safety to Wratten safelights of untreated film and film desensitised for varying times with varying concentrations of Pinakryptol Green was studied. By bathing panchromatic film in 1 : 10,000 Pinakryptol Green, or after it has been in a developer containing 1 : 25,000 of the dye for 2 or 3 minutes, the film may be safely inspected with a Series 4 Wratten safelight containing a 25-watt lamp at a distance of 1 ft. Under similar conditions motion-picture negative film may be safely examined with a Series 0 safelight. A latent image on a desensitised emulsion tends to bleach when exposed to red light. The bleaching is most marked with non-colour-sensitive emulsions; with panchromatic emulsions it is not serious, and after development has commenced no appreciable bleaching occurs. With desensitised non-colour-sensitive emulsions the safe time of exposure to a red safelight is determined by the time required to destroy the latent image, and not the time

required to produce fog. Data are given for the fogging action of desensitisers with a number of developers, and an exhaustive study is recorded of the effect of Pinakryptol Green used in the developer instead of as a preliminary bath. No desensitiser is known which is stable in a developer rich in quinol. Desensitisers affect the rate of development to an extent which varies with the developer and with the dye used. W. Clark.

Production of photographic images with diazo-compounds. M. Battegay and P. Braun (Bull. Soc. Ind. Mulhouse, 1926, **92**, 274—276).—Paper is bathed in a solution of Diazo Yellow Light G or 2 G (Bayer), washed, diazotised in the dark in a solution of sodium nitrite acidified with hydrochloric acid, and dried at a low temperature. Printing is carried out under a negative in the shade, and the back of the paper is given a uniform exposure to prevent its coloration in the subsequent processes. After exposure the print is treated in a bath containing a coupling agent, the most satisfactory of which are resorcinol (for yellow), phenylmethylpyrazolone (red-purple), α-naphthylamine (deep violet), chromotropic acid (garnet), and H-acid (violet). The stability to light of the images is further increased by bathing in copper sulphate solution, or basic dyes such as Methylene Blue or Malachite Green. W. Clark.

Fifty years of photography. C. E. K. Mees (Ind. Eng. Chem., 1926, **18**, 915—916).

Patents.

Photographic film and method of marking it. E. Taylor and E. F. Chandler, Assrs. to Taylor Laboratories, Inc. (U.S.P. 1,590,597, 29.6.26. Appl., 17.6.24).—A cellulose film prepared as described in U.S.P. 1,590,593 (p. 818) is impregnated with a sensitive silver salt for photographic purposes. For instance, to sensitise the film it is impregnated on one side with silver nitrate solution and then exposed to the action of a halogen. The silver halide is formed inside the film so that scratching of the latter is not injurious to the photograph. R. B. Clarke.

Production of photographic gelatin reliefs. S. de Procoudine-Gorsky and N. Pozniakow (E.P. 243,338, 10.11.25. Conv., 19.11.24).—See F.P. 589,422; B., 1926, 220.

XXII.—EXPLOSIVES; MATCHES.

Laws of combustion of colloidal powders. II. H. Muraour (Bull. Soc. chim., 1926, [iv], **39**, 1115—1119).—Detailed results are given in support of the generalisations previously stated (B., 1926, 722), and showing that nitroglycerin powders having very different explosion temperatures obey the same laws of combustion. R. Cuthill.

Explosives, 1876—1926. H. Schlatter (Ind. Eng. Chem., 1926, **18**, 905—907).

Patents.

Blasting-powder composition. C. D. Pratt, Assr. to Atlas Powder Co. (U.S.P. 1,590,393, 29.6.26. Appl., 4.6.25).—The addition of 5—35% of a carbohydrate, such as starch or cellulose, and of 1—15% of an inorganic chloride, such as ammonium chloride, sodium chloride, or barium chloride, to black blasting powder yields a cool explosive which may be safely used in gassy or dusty mines. T. S. Wheeler.

Making smokeless powder and the like. E. Taylor, Assr. to Taylor Laboratories, Inc. (U.S.P. 1,590,598, 29.6.26. Appl., 17.6.24).—Cellulose obtained as described in U.S.P. 1,590,594 (p. 818) is partially dried after leaving the washing tank and granulated in the manner usually adopted for smokeless powders. If a dense powder is desired, the grains are thrown, in a continuous stream, into a mixture of nitric and sulphuric acids. After nitration they are placed in a vessel, perforated at the bottom, and the nitrating mixture is removed by displacement with weak acid. The product is then centrifuged, boiled with water, and dried. The smokeless powder thus obtained consists of tough, dense grains of glass-like appearance. R. B. Clarke.

Dynamite composition. W. R. Swint, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,594,861, 3.8.26. Appl., 13.4.22).—An explosive of low density contains about 7—15% of a normally liquid high explosive, about 5—15% of finely divided balsa wood, and ammonium nitrate. S. Binning.

XXIII.—SANITATION; WATER PURIFICATION.

Chlorine treatment of water and sewage. G. Ornstein (Z. angew. Chem., 1926, **39**, 1035—1037).—A résumé of the work carried out in Germany since 1920, fully confirming the advantages of the treatment. S. I. Levy.

Clinical and laboratory investigation of the effect of metallic zinc, of zinc oxide, and of zinc sulphide upon the health of workmen. R. P. Batchelor, J. W. Fehnel, R. M. Thomson, and K. R. Drinker (J. Ind. Hyg., 1926, **8**, 322—363).—Detailed clinical and laboratory studies on 24 workmen of the New Jersey Zinc Co. over periods varying from 2 to 35 years, during which they were exposed to the inhalation of fumes of metallic zinc, zinc oxide, and zinc sulphide produced from ores containing only very small proportions of lead, cadmium, and arsenic, have shown that no chronic or acute illnesses follow prolonged exposure to zinc fumes, and that all diseases previously ascribed to zinc poisoning must be due to the presence of toxic impurities, such as lead, cadmium, arsenic, and antimony, which are usually associated with zinc ores. Zinc workers absorb considerable amounts of zinc, but practically all of it is excreted, although the blood contains a slightly higher content than the normal. This absorption and excretion of zinc can go on for many years without causing any affection of the kidneys, stomach, or intestines, and has no effect on the metabolism of the living cells. A. R. Powell.

Water supply and sewerage during the past fifty years. R. S. Weston (Ind. Eng. Chem., 1926, **18**, 899—901).

Patents.

Heating and de-aerating boiler feed and other water. Cochrane Corp., Assees. of G. H. Gibson (E.P. 240,417, 5.8.25. Conv., 26.9.24).

Tricalcium cyanide (U.S.P. 1,592,127).—See VII.

Insecticides from *Derris* species (E.P. 247,140).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCTOBER 29, 1926.

I.—GENERAL; PLANT; MACHINERY.

Utilisation of diphenyl oxide in power generation. H. H. Dow (Mech. Eng., August, 1926; Chem. Met. Eng., 1926, **33**, 475—476).—Diphenyl oxide may be used as a material of high b.p. in a bi-fluid system of power generation, *e.g.*, as a substitute for mercury in mercury vapour–steam boilers, in which mercury vapour is used to drive a turbine and the condensed mercury produces steam in a boiler. Diphenyl oxide can be produced in unlimited quantities at a cost per unit volume of less than 2% that of mercury; it is quite stable at the usual operating pressures and temperatures of the boiler. The weight of diphenyl oxide vapour is about 9·4 times that of steam, and for the same horse-power a diphenyl oxide vapour turbine will run at a lower speed with higher torque, which may result in increased turbine efficiency. One disadvantage is that diphenyl oxide vapour superheats when it expands adiabatically in the turbine, and it is difficult to transfer heat from a superheated vapour; to get maximum efficiency, it is therefore necessary to use this exhaust superheat for a regenerative heating of the boiler feed. The use of diphenyl oxide has considerable commercial possibilities where coal is dear. B. W. Clarke.

Radiation pyrometers. A. J. Philpot (J. Sci. Instr., 1926, **3**, 366—373).—The effect of varying the distance from the radiating source upon the indications of total radiation pyrometers of the focusing and non-focusing lens and mirror types is discussed theoretically and practically. J. S. G. Thomas.

Effect of acetone as an impurity in spirit thermometers. W. F. Higgins (J. Sci. Instr., 1926, **3**, 361—365).—In the case of spirit thermometers containing acetone as an impurity in the filling liquid, a marked depression of the reading corresponding to a definite temperature is obtained over a period of some years when the thermometers are exposed to light. This effect is attributed to contraction of the liquid consequent upon the formation of condensation products from the acetone under the influence of light. Commercial methylated spirit commonly contains acetone, and it is shown that such spirit, either in the commercial or distilled form, is unsuitable for the construction of spirit thermometers. Pure ethyl alcohol, acetone-free methyl alcohol, or a mixture of these two substances is suitable. J. S. G. Thomas.

Influence of segregation on corrosion of boiler tubes and superheaters. G. R. Woodvine and A. L. Roberts (J. Iron and Steel Inst., 1926, **113**, 219—228).—See B., 1926, 471.

Adsorbent carbons. Honig.—See II.

Patents.

Utilisation of heat in evaporation processes. W. A. Gilchrist (E.P. 229,649, 2.2.25. Conv., 19.2.24). —Steam at a high primary pressure (*e.g.*, 100—200 lb./sq. in.) and superheat (*e.g.*, up to 110°) is utilised in a prime mover, and exhausted to a pre-evaporator when still at a fairly high secondary pressure (*e.g.*, 15 to 100 lb./sq. in.) and temperature (up to 55° superheat). The secondary temperature is regulated by mixing with primary steam, which is by-passed direct from the boiler and/or by the use of a humidifier, to suit the maximum temperature that the liquid to be evaporated will withstand. The gases are circulated rapidly in the pre-evaporator to prevent a rise of temperature of the liquid above 113—116°; the heating vapour is supplied at a superheat of 23—28° in the case of sugar juice, and the vapour from the juice or tertiary steam at a pressure of 8—10 lb./sq. in. is used in a multiple-effect evaporator. Various other refinements are claimed. B. M. Venables.

Evaporators. Griscom-Russell Co., Assees. of S. Brown (E.P. 236,901, 20.5.25. Conv., 14.7.24).—Heating elements for use in scale-forming liquors are constructed of double-spiral tube coils, with the plane of the coils horizontal, the tube being bent back on itself so that all convolutions are in the same plane and both inlet and outlet are outer ends of the coil. The tube is flattened with the long axis of the section vertical, so that it distorts considerably (like a Bourdon tube) when pressure (steam) is applied to the interior; also more space is left between coils, compared with a circular tube, both effects helping to prevent accumulation of scale. B. M. Venables.

Refrigerating apparatus of the absorption type. A. L. Mond. From Platen-Munters Refrigerating System Aktiebolag (E.P. 239,545, 28.3.25).—In a refrigerating apparatus using an inert carrier gas, the absorber is so constructed that it serves also as a cooler for the boiled-off liquid, this cooling being additional to the usual heat interchange between the liquid passing to and from the boiler. Part of the cooling is effected in the upper part before the liquid is allowed to mix with the gas to be absorbed in the lower part of the absorber. B. M. Venables.

Refrigerating apparatus working on the absorption principle. Mannesmann Kälte-Ind. A.-G. (E.P. 245,075, 6.11.25. Conv., 24.12.24).—The invention relates to the supply and control of cooling water to the boiler-absorber and to the condenser of an absorption refrigerator. The water passes through a rocking distributor, which is operated by changes of temperature in some part of the apparatus, *e.g.*, the boiler, and is diverted alternately through straight, easily cleaned tubes in the boiler

or to the lower part of a vessel of water surrounding the condensing coil. A separating vessel for removing absorbent (*e.g.*, water) from the vapour of the refrigerant (*e.g.*, ammonia) is also partly submerged in the latter body of water. Provision is made that in the event of any undue rise in temperature the rocking distributor will send the cooling water through the boiler. A device for indicating undue rise in pressure of the cooling water supply consists of a tube by-passing the distributor and provided with a small pinhole out of which water will squirt should the pressure rise above normal.

B. M. VENABLES.

Pulverising mills. RAYMOND BROS. IMPACT PULVERIZER Co., Assees. of C. M. LAURITZEN and W. H. VOGEL (E.P. 248,347, 10.2.26. Conv. 2.3.25).—A disintegrator is provided with a deflecting ring or fillet in the corner of the casing to return material into the path of the beaters. The ring is preferably adjustable from outside so as to be set as close as possible to the beaters.

B. M. VENABLES.

Liners for tube and like mills. H. A. GILL. From F. L. SMIDTH & Co. A.-S. (E.P. 257,218, 6.7.26).—A liner for tube mills that will not wear smooth is constructed of bars of metal or other hard material (preferably connected by cross-pieces) interspaced and held in place by softer material such as cement grout. The bars are originally set so as to protrude above the softer material, and will wear continuously in that state until practically nothing is left. B. M. VENABLES.

Pulverising device. W. S. MORTON (U.S.P. 1,597,656, 24.8.26. Appl., 21.8.25).—The material to be ground is impelled at high velocity by such means as jets of fluid, from opposite (or several) sides of a chamber against grinding surfaces of silica, the angles of impact being so arranged that the various streams after rebound interfere with each other. B. M. VENABLES.

Reducing solid bodies to an extremely fine state of division and the preparation of colloidal solutions. S. G. S. DICKER. From ODERBERGER CHEM. WERKE A.-G. (E.P. 256,094, 30.10.25).—The pulp to be ground is pumped very slowly tangentially into a mill in which beaters are rotating at high speed so that the relative motion between the beaters and liquid is very great. The exit conduit is also tangential to the mill, and falls away therefrom in a curve exactly corresponding to the parabolic fall of the material if it were unrestrained. The operation is generally intermittent, the material being pumped from a storage vessel through the mill back to the storage vessel until the batch is finished. B. M. VENABLES.

Filtering apparatus. J. M. PHILIPPS (E.P. 255,960 and 256,309, 2.5.25).—(A) A filter where the filtering medium is a solid porous block or pack of leaves is provided with one set of long holes or passages to which the pre-filt is supplied and in which the solids collect, and another set of holes from which the filtrate flows out. The collected residue is discharged by a number of rods pushed down into the first set of holes, but the further end of these holes is kept closed by an "obturator" slide until the residue has been compressed by the descent of the rods, so that when the discharge ends are opened by sliding the obturator plate the residue is pushed out as firm and dry cakes. The rods are supported by a piston which may be driven by the pressure of the pre-filt itself. Automatic means are provided to work the various valves and the obturator, so that continuous working may be maintained. (B) After discharging the residue as above, the filter is cleaned by a back flow of liquid or gas admitted under pressure into the passages for filtrate. B. M. VENABLES.

Filtering apparatus. G. AZZOPARDI (E.P. 256,124, 12.1.26).—An apparatus for filtering oils, waxes, syrups, etc. comprises a vessel into the upper part of which the material to be filtered is charged, and in which it is heated by a steam coil and placed under air pressure by a self-contained or separate pump. The liquid first passes through loose material such as cotton-wool, then through closer material such as layers of felt—interleaved, if desired, with paper—and finally through a linen bag-like diaphragm. The last-named will serve to contain any decolorising or reacting material that may be necessary. The filtered liquid then falls to the lowest part of the apparatus and passes upwards over a baffle to effect separation of water. B. M. VENABLES.

Filter. F. B. LOMAX (U.S.P. 1,595,344—5, 10.8.26. Appl., [A] 31.1.24, [B] 29.9.24).—(A) Enclosed spaces above and below a filtering barrier are filled respectively with gas and with liquid, and the gas-filled space is connected to a supply of liquid. Means are provided for withdrawing liquid from the liquid-filled space to draw gas through the barrier, thus producing a vacuum for suction of the liquid from the supply. (B) An upper cup telescopes into a lower cup, and both are mounted on a frame, including a stand with a top portion. The lower cup is provided with lugs connected to the stand through the top portion, and a yoke secured to the top portion straddles the cups. A partition is placed between the cups, and filtering elements between the partition and the cups. An inlet extends upwards through the lower cup and the partition, and communicates with the spaces above the filtering elements, and means are provided on the yoke for engaging the upper cup to exert pressure on the elements and the partition against the lower cup. H. HOLMES.

Multiple-chamber filter press. P. W. PRUTZMAN, Assr. to GEN. PETROLEUM CORP. (U.S.P. 1,595,616, 10.8.26. Appl., 29.3.22).—Separate flanged tubular members, consecutively arranged and permanently assembled, are provided, and a non-perforated plate with a corrugated filter-supporting surface is disposed between each pair of adjacent members. H. HOLMES.

Centrifugal separators. AKTIEBOLAGET SEPARATOR (E.P. 249,850, 16.3.26. Conv., 26.3.25).—A centrifugal bowl in which the separated solids are allowed to accumulate has the inlet and outlet for liquid both at the top, and is provided with means to remove the residual liquid when stopping for cleaning, consisting of a siphon pipe or pipes of which the outlets pass downwards near the axis, and of which the inlets are formed as channels between the bottom of the bowl and the distributing plate next above. B. M. VENABLES.

Imparting to desiccants a large superficial area. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (E.P.

253,114, 1.6.26. Conv., 8.6.25).—The desiccant, *e.g.*, phosphorus pentoxide, is deposited upon a pumice, glass wool, or other porous carrier, a liquid hydrate of the desiccant, *e.g.*, metaphosphoric acid, being first applied to the carrier to serve as a binder. H. Holmes.

Heating the blast for furnaces, producers, and the like. J. Y. Johnson. From Badische Anilin & Soda Fabrik (E.P. 255,281, 10.10.25).—Air is preheated for producers and furnaces by atomising in the blast pure or crude tar by means of nozzles, and burning it in the blast prior to its entrance into the producer or furnace. A. C. Monkhouse.

Rotary furnaces for molten material. G. de Bethune (E.P. 256,158, 30.3.25).—The wall of a rotary furnace is cooled by means of an open chamber at the extreme end portion of the metal casing, into which water is projected laterally by means of a large number of jets around the furnace. The inner face of the metal casing is provided with retaining ribs for holding the refractory facing in position, and subsequently for damming the flow of molten material and facilitating its solidification. M. Cook.

Kilns. G. Balz (E.P. 256,547, 20.5.26. Addn. to 245,348).—In the kiln or roaster described in the original patent (cf. B., 1926, 196) strickles as well as rakes are provided, depending from the roofs of the various hearths to level the material and permit cooling air to circulate freely. The cooling air is admitted through a vertical perforated cylinder situated on the axis of the furnace; the air leaves this distributor horizontally and passes partly over the material on the hearth and partly through the material dropping through the port from the hearth above. The air on its way to the distributor may pass through several alternative pipes led through parts of the furnace which are at different temperatures, so that the temperature of the air may be regulated. B. M. Venables.

Furnace for [high-temperature] chemical reactions. L. Hamburger and E. C. Prins, Assrs. to N. V. Stikstofbindingsind. "Nederland" (U.S.P. 1,590,161, 22.6.26. Appl., 15.2.24. Conv., 13.3.23).—The walls of the furnace consist of a thin outer wall of refractory material and an inner wall of solid metal, between which circulates a molten metal containing in solution or suspension a reducing agent such as carbon. The furnace is designed for all reactions where the mechanical wear on the furnace walls is such as to require great strength, although it may be adapted for internal combustion. The furnace is specially useful for alkaline reactions, such as the manufacture of cyanide by heating an alkaline-earth carbonate in an atmosphere of nitrogen. E. S. Kreis.

Apparatus for effecting intimate contact between liquids and gases. Kirkham, Hulett & Chandler, Ltd., and W. F. Slater (E.P. [a] 256,351, 25.5.25, and [b] 256,358, 2.6.25).—(a) A casing is divided into a number of superposed compartments in each of which is rotated by a common shaft a spraying tray with vertical perforated walls which extend practically the full height of each compartment. The liquid passes downwards through the compartments in succession, while the gas passes upwards, the transfer from compartment to compartment being through segmental openings, which are provided with lips to retain a layer of liquid, and with inner walls to prevent the sprayed liquid passing down. In one case successive openings are diametrically opposite to each other, and the gas passes both ways around a compartment before passing up into the next; alternatively, the openings may be nearly or exactly over each other, in which case all the gas passes around the compartment in one direction. Spiral baffles may be provided to prevent short-circuiting. (b) In a sprayer comprising a vertical perforated cylinder, lifting vanes are provided in the annular space between the cylinder and the shaft to pick up the liquid, and guides in the shape of inverted truncated cones are provided to distribute the liquid over the perforated spraying surface in a density that may be uniform or varying as desired. B. M. Venables.

Apparatus for separating finely-divided solids from liquids. [Thickeners.] J. S. Withers. From J. W. Wickes (E.P. 256,995, 19.2.25).—The apparatus comprises a number of superposed thickening trays. Rakes deliver the settled material to a common peripheral or central discharge, and clear liquid is drawn off through pipes from the highest point of each settling space, also, if desired, from the highest point of the containing tank. B. M. Venables.

Catalysing apparatus. D. A. Legg and C. W. Hancock, Assrs. to Commercial Solvents Corp. (U.S.P. 1,580,740, 13.4.26. Appl., 20.2.23; cf. E.P. 166,249; B., 1921, 614 a).—An apparatus for working catalytic reactions at a constant temperature consists of an annular catalyst chamber formed by two vertical concentric cylinders immersed in a bath maintained at a constant temperature. The material to be treated passes down through a conduit also immersed in the bath and up through the catalyst. The cylinder forming the inner wall of the catalyst chamber is removable to permit easy access to the catalyst. The apparatus is useful for the dehydrogenation of *n*-butyl alcohol to butaldehyde using the catalyst described in E.P. 166,249 (*loc. cit.*). T. S. Wheeler.

Process of deflocculating solids [graphite]. G. W. Acheson (U.S.P. 1,595,052, 3.8.26. Appl., 8.3.26).—A small proportion of an organic deflocculating agent, *e.g.*, tannin, together with aqueous ammonia, is added to graphite. The paste is subjected to attrition until the tannin has been adsorbed; the process is repeated a number of times, sufficient tannin being added to cause reflocculation on thorough mixing. The reflocculated graphite is next deflocculated by addition of infusions of grass under the same conditions as before. In this way the ultimate yield of colloid and its rate of preparation are considerably increased. R. B. Clarke.

Condensing system. E. J. Atckison, Assr. to Southwestern Condenser Co. (U.S.P. 1,595,334, 10.8.26. Appl., 1.11.22).—Mixed vapours are condensed fractionally in a series of condenser units, the vapour from one unit being fed to the next one. Each unit comprises a number of cooling tube divisions, connected in series or in multiple. A common cooling system passes cooling fluid through the units in series. H. Moore.

Distillation columns. W. ROTHENBACH (G.P. 430,622, 12.4.25. Conv., 12.4.24).—The steam or vapour passages of a distillation column are so arranged that a rectangular space remains for the introduction of one or more heating elements through cleaning openings; the elements are shaped so that they can be easily introduced through the openings, and they are fastened to the cleaning covers. The sections of the column are in the form of segments of a circle, so that the heating elements can be inserted easily, and the remaining space of the column utilised to the best advantage.
W. G. CAREY.

Manufacturing finely-divided solid substances. V. KOHLSCHÜTTER (U.S.P. 1,596,979, 24.8.26. Appl., 30.8.21. Conv., 4.8.18).—The substances are vaporised, introduced into a gaseous medium, and precipitated electrically.
H. HOLMES.

Fireproofing organic fibrous material. [Preparation of filtering material, catalyst carriers, etc.] H. STELLING (G.P. 429,918, 10.4.24).—Finely-divided silica is deposited throughout the material by subjecting it in the presence of moisture to an atmosphere of silicon chloride. A product suitable for use, *e.g.*, for filtering liquids, for purifying and drying air, or as a catalyst carrier, is obtained by cautiously burning away the organic material so that the residue retains the original structure.
L. A. COLES.

Gas washers, absorption apparatus, etc. (E.P. 257,208—9).—See II.

Density regulator for evaporators (U.S.P. 1,595,244).—See VII.

Precipitating suspended particles from gases (G.P. 431,216).—See XI.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Coal research. [Oxidation and reduction.] F. FISCHER (Abhandl. Kennt. Kohle, 1925, **7**, 257—267; Chem. Zentr., 1926, II, 1480).—The complete chemical utilisation of coal may be attained either by regulated oxidation with oxygen or air (ozonisation, oxidation under pressure), or by reduction with agents such as hydrogen or carbon monoxide. By the former method, all sorts of coals may be completely converted into soluble organic compounds, of which only a fraction has so far been identified, with a yield of 50% by wt. of the coal. Lean coals, for example, yield 40% of non-volatile organic acids, of which more than a quarter have been identified, and amongst which are about 12% (of the wt. of coal taken) of a mixture of benzoic and phthalic acids. Other benzenecarboxylic acids, in particular the pentacarboxylic acid, may be obtained by varying the experimental conditions. In considering the action of hydrogen on coals particular prominence has been given to the conversion of the latter into synthetic mineral oils and light motor fuels. There appears to be some doubt, however, as to whether the treatment of solid, *i.e.*, non-pumpable, fuels under pressure, for the production of petroleum-like substances, will be feasible on a large scale owing to technical difficulties. It may be assumed that low-temperature carbonisation, and the conversion of the primary tar into light motor fuels, has the greatest prospect of realisation in the near future.
W. T. K. BRAUNHOLTZ.

Artificial production of coal from cellulose and lignin in the presence of water. H. TROPSCH and A. VON PHILIPPOVICH (Abhandl. Kennt. Kohle, 1925, **7**, 84—102; Chem. Zentr., 1926, II, 1482).—The artificial production of coal, described by Bergius, is often taken as evidence of the origin of coal from cellulose. Repetition of Bergius' experiments shows that artificial coal formation from cellulose, lignin, and wood in the presence of water yields not only substances insoluble in water, but also a considerable quantity of water-soluble products and volatile acidic and neutral compounds. Bergius makes no mention of water-soluble products obtained from cellulose, nor of substances extractable with ether, and the formation from cellulose of these water-soluble compounds at the same time as charred matter is neglected in his theoretical discussion of experimental results. An average yield of 30% of artificial coal was actually obtained from cellulose as against a yield of 46%, as deduced from Bergius' equation, and this yield was further influenced to a large extent by the nature of the material of which the autoclave was made.
W. T. K. BRAUNHOLTZ.

Humic substances in lignite. P. KAUNERT (Braunkohlenarchiv, 1926, **10**, 39—101; Chem. Zentr., 1926, II, 674—675).—Humic acids extracted from lignite are not identical with the humic substances as they exist in the lignite. The nature of the extract depends upon the proportion of humins to the coal substance and upon the conditions of extraction. Lignites were extracted with dilute alkali solution at 65° and the amount of soluble extract, determined colorimetrically, was plotted against the time of extraction. The form of curve showed that the reaction was of the second order, and probably a saponification. Lignite contains only very small quantities of free humic acid. Extraction under increased temperature and pressure results in a further breakdown of the coal substance and an increased quantity of extract.
S. PEXTON.

Coking constituents of Mesa Verda and Pittsburgh coals. J. D. DAVIS and D. A. REYNOLDS (Ind. Eng. Chem., 1926, **18**, 838—841).—Solid and oily bitumens, extracted from Mesa Verda (Utah) non-coking coal and Pittsburgh coking coal by means of benzene under pressure and separated with light petroleum, were mixed separately with the extracted coal residue and with coke, and the mixtures were carbonised in a crucible, as in the test for volatile matter. Contrary to the findings of Fischer and in agreement with those of Bone, the oily bitumen had little agglutinating power, whilst that of the solid bitumen was strong. Extraction with benzene did not remove all the bitumen from the coals, and β- and γ-compounds extracted from the coal residue with pyridine possessed agglutinating power. The bitumens of the Utah coal, unlike those of the Pittsburgh coal, distil at low temperatures without extensive decomposition, and this is probably responsible for the non-coking nature of the former coal. The suitability of the test for volatile matter for estimating the coking properties of coal constituents is questioned, as the

conditions of carbonisation are widely different from those obtaining in ovens and retorts.

W. T. K. Braunholtz.

Low-temperature carbonisation of coal. I—III. A. C. Fieldner (Fuel, 1926, 5, 203—214, 265—271, 294—297).—Different processes of low-temperature carbonisation are described and a division is made into processes using externally-heated and internally-heated retorts respectively, and two-stage carbonisation processes. The internally-heated retorts are simpler, but are limited to non-caking, feebly-caking, or briquetted coals; the gas produced is of low calorific value, and the light oils are not recoverable from the diluted gas. Externally-heated retorts are more suitable for the manufacture of a domestic fuel from poorly-caking coal, and yield a rich gas and light oil. Low-temperature carbonisation in the Central and Western States of America, where little coking coal exists, affords a method of competing with the high-temperature processes requiring more expensive raw material. A. C. Monkhouse.

Comparative distillations of cellulose, lignin, and deresinified wood under diminished pressure. F. Fischer and H. Tropsch (Abhandl. Kennt. Kohle, 1925, 7, 181—186; Chem. Zentr., 1926, II, 1482).—Cellulose gave about 43% of a water-soluble distillate, which was optically active and contained lævoglucosan. The water-soluble portion of the wood tar was also optically active, but that from lignin was not, showing the absence of lævoglucosan. Wood is intermediate between cellulose and lignin as regards yield of tar and coke and the nature of its distillation products. Like lignin, it yields alkali-soluble, phenolic distillation products. The phenols in lignin tar obtained under diminished pressure are not due to changes brought about in the treatment of the lignin with hydrochloric acid.

W. T. K. Braunholtz.

Treatment of wood charcoal with water under pressure and at high temperatures. H. Tropsch (Abhandl. Kennt. Kohle, 1925, 7, 107—110; Chem. Zentr., 1926, II, 1482).—The reaction between wood charcoal and water does not simply follow the water-gas equation, but if charcoal that has been heated to redness is then treated with water at a lower temperature, considerable quantities of methane are formed. Wood charcoal that has been subjected to a high temperature gives only a small amount of methane when heated with water under pressure. W. T. K. Braunholtz.

Comparison of adsorption carbons. P. Honig (Koll. Chem. Beihefte, 1926, 22, 345—420).—Charcoal made by heating a mixture of blood with 10% or 20% of potassium carbonate at above 750° and then treating with hydrochloric acid is more active than the product obtained by using calcium phosphate instead of potassium carbonate. The presence of nitrogen is not essential, as substitution of sugar for blood does not impair the activity. Carbonisation of fir-wood sawdust impregnated with zinc chloride gives a charcoal closely resembling "carboraffin." If ordinary charcoal is heated in a vacuum, or out of contact with oxidising gases, its activity is increased only slightly or not at all. Activation by heating in a current of steam yields a carbon very similar to "norit," the activity being the greater the larger the quantity of steam used. In this process the charcoal loses hydrogen. Activation by carbon dioxide should be carried out at about 1000°, and the charcoal should be as finely divided as possible. "Supernorit" is apparently made by activating wood-charcoal with furnace-gases in this manner. In general, there is no relation between the purity of the inactive charcoal and the activity of the product obtained from it. The activated carbons prepared as described above are very complex in composition, but in general contain less hydrogen than ordinary wood charcoal. Bases are readily adsorbed and acids to a slightly less extent, whilst neutral salts of the alkali metals are not adsorbed at all. The order of the adsorptive powers of the various carbons for a given substance varies with the substance used, but, in general, substances with relatively small molecules, such as iodine, are adsorbed to a much greater extent than those with larger molecules, *e.g.*, quinine, this being probably due to the fineness of the capillaries. The decolorisation of sugar solutions by activated carbons depends on the p_H of the solution and the nature of the impurities present, and is hindered by adsorption of the sugar itself. The densities of activated carbons are greater than that of ordinary charcoal, and, with steam-activated varieties, increase with the length of the treatment with steam; but there is no connexion between the density and the adsorptive power. The heat of wetting by organic liquids, however, runs closely parallel to the adsorptive power, and a determination of this value therefore gives a good indication of the technical value of the carbon. No direct connexion appears to exist between the reactivity with sulphur, sulphuric acid, or nitric acid and the adsorptive power. Heating at 1000° in an atmosphere of nitrogen reduces the activity of carboraffin by about one half, but has little effect on the other carbons. Wood charcoal and steam-activated carbon cause practically no decomposition of sugar solutions, but carboraffin causes inversion to a small extent. R. Cuthill.

Increasing the calorific value of combustible gases by the decomposition of tar vapours. H. Strache (Z. Ver. Gas- u. Wasserfachmänner Oest., 1926, 64, 111—116; Chem. Zentr., 1926, II, 1355—1356).—In the usual process for the production of water-gas, tar vapours are decomposed by the high temperature and the action of undecomposed steam into carbon dioxide and hydrogen, and there is no appreciable increase in the calorific value of the gas. In a process devised by the author the tar vapours are separated from the coal at a predetermined temperature, and then returned continuously into a zone of higher temperature. The calorific value of the gas is increased in this way by about 400 cal. L. A. Coles.

Sulphur content of mixed gas after preheating in the regenerators of a Siemens–Martin furnace. E. Will and W. Hulsbruch (Mitt. Versuchsanst. Deuts.-Luxemburg. Bergw.- u. Hütten-A.-G., 1925, 1, 242—247; Chem. Zentr., 1926, II, 674).—The sulphur content of mixed blast-furnace and coke-oven gas is reduced by 25—30% during its passage through the regenerators of a Siemens–Martin furnace. Only 2·5—4% of the hydrogen sulphide and perhaps the small content of

carbon disulphide are removed by thermal dissociation, the bulk being taken up by the refractory of the regenerators. When the regenerators are reversed the accumulated sulphur is oxidised and passes away with the waste gases. S. Pexton.

Higher-boiling constituents of "synthol." H. Tropsch (Abhandl. Kennt. Kohle, 1925, **7**, 75—77; Chem. Zentr., 1926, II, 1483).—The higher-boiling constituents of "synthol" contain small quantities of viscous oils, which can be acetylated and therefore contain hydroxyl groups. The least volatile constituents are solid and asphaltic. Traces of a solid, paraffinic substance are also obtained. W. T. K. Braunholtz.

Determination of the calorific power of benzine. O. Cantoni (Giorn. Chim. Ind. Appl., 1926, **8**, 119).—In determining the calorific value of petrol, especially aviation petrol, the liquid cannot be weighed satisfactorily in the ordinary open crucible of the Mahler bomb, even when a layer of kieselguhr or the like is used. Introduction of the liquid into the bomb in a sealed glass bulb is also inexpedient, since if the point of the bulb is broken at the moment the bomb is closed, ignition of the liquid by the wire seldom occurs, whilst rupture of the bulb in the closed bomb by the pressure of the oxygen often results in the formation of a detonating gaseous mixture. A convenient plan is to weigh the petrol in a small cylindrical gelatin capsule such as is used by pharmacists, the igniting wire being passed through two needle holes in the lid of the capsule, so that it can be hung from the hook of the balance pan. The capsules should be stored in a closed vessel to keep their moisture content constant, and allowance must be made for their mean calorific value, this being determined beforehand. With (1) benzene, and (2) toluene, for which Landolt and Börnstein's tables give the calorific values, 10,025 and 10,160, respectively, the values obtained by the above procedure were (1) 10,038 and 10,052, and (2) 10,151 and 10,172. Three determinations with a sample of aviation petrol gave the values, 11,034, 11,005, and 11,021 cal. T. H. Pope.

Increase in the viscosity of oils subjected to a silent discharge. H. Becker (Wiss. Veröff. Siemens-Konz., 1926, **5**, 160—166).—When mineral or fatty oils are subjected to a silent discharge an increase in viscosity takes place. With saturated hydrocarbons this is accompanied by evolution of hydrogen. The increased viscosity is attributed to the formation of new compounds by the union of 2 molecules which have lost hydrogen atoms. The method may be used for the preparation of hydrogen of exceptional purity. Hydrogen can also be obtained from inorganic compounds by similar treatment, and new compounds are formed by the molecular residues. C. J. Smithells.

Examination of some transformer oils after long exposure to light. H. von der Heyden and K. Typke (Petroleum, 1926, **22**, 1024—1025).—Seven transformer oils formed a sludge after exposure to light in closed flasks for $1\frac{1}{2}$ years and developed a strong odour of the lower fatty acids. An increase occurred in the tar and acid values, this being especially marked in two American oils which had not been refined with sulphuric acid, and in a well-refined Russian oil. Three regenerated oils formed only a trace of sludge, with slight deterioration. An American non-sludging oil decreased in tar value from 2·1 to 0·08, and in acid value on the blown oil from 2·1 to 0·07, similar decreases being observed by preserving this oil out of contact with light for a similar period. Attention is drawn to the danger of storage or transport of transformer oils in light glass vessels, this being the cause of many discrepancies in the analyses of oil samples. E. H. Sharples.

Determination of the formolite value. A. Nastjukov (J. Chem. Ind. [Russ.], 1925, **1**, [4], 28—30; Chem. Zentr., 1926, II, 947).—Dry easily washed formolites are obtained by the following procedure: 1 pt. of the oil is shaken with 4 pts. of sulphuric acid and 2 pts. of formalin, the mixture is heated for 1 hour on the water-bath under a reflux condenser, and then poured into five times its volume of water. If an oil layer separates it is pipetted off. The remainder is boiled for 1 hour with steam, treated with ammonia, filtered through a suction filter, and the residue washed with water and benzine and dried at 100°. The weight of the dried formolite must be increased by 5·25% to obtain results comparable with those obtained by earlier investigators, in which the formolites were weighed air-dry. The dried formolites lose one-third to one-quarter of their weight when subjected to renewed treatment with benzene or petroleum spirit.

Determination of the formolite value. A. Nastjukov (Neftjanoe Chozjajstvo, 1926, **10**, 513—515; Chem. Zentr., 1926, II, 1206).—Considerable quantities of adsorbed oil are extracted with benzene or petroleum ether from formolites prepared as described by the author (cf. preceding abstract). The true formolite value should be calculated from the weight of the residue left after extracting the oil, and not from the weight of the crude material. The adsorbed oil has a lower viscosity than the original oil, and consists entirely of naphthenes, which do not react with formaldehyde. The presence of naphthenes, therefore, does not increase the viscosity of the oil. In the case of cylinder oils the higher the viscosity the greater is the formolite value. L. A. Coles.

Coal blending. D. Brownlie (J. Iron and Steel Inst., 1926, **113**, 229—283).—See B., 1926, 650.

Distillation of coal with superheated steam. Dunkel.—See III.

Lubricants from tar products. Spilker.—See III.

Condensation of methyl alcohol. Tropsch and von Philippovich.—See XX.

Patents.

Briquettes from pulverised fuel. K. H. R. Tillberg (E.P. 249,061, 6.5.25. Conv., 11.3.25).—Briquettes of pulverised fuel, bonded with molasses which has been previously caramelised by heating to 200°, are stable to water, and do not disintegrate when heated suddenly. S. Pexton.

[Fuel] briquette and process. J. P. Delzeit, Assr. to Lehigh Coal and Navigation Co. (U.S.P.

1,596,239, 17.8.26. Appl., 4.12.25).—Fine coal is bonded with sulphite liquor and a relatively small proportion of free sulphur and the product baked at 320—370°.
S. Pexton.

Briquetting of coal dust in stages by pressure. F. Hofmann, M. Dunkel, M. Heyn, and W. Grote (G.P. 431,502, 9.9.24).—Coal dust is first compressed at a low temperature (up to about 350°), and the briquettes so formed are finally compressed at a higher temperature. The process completely eliminates the risk of explosion and requires much lower temperatures and pressures than the usual methods. W. T. K. Braunholtz.

Treatment of peat for removal of water. T. Rigby (E.P. 256,327, 7 and 12.5.25).—The water content of peat can be more efficiently reduced by pressure if the peat has been preheated. The presence of earth and stony materials renders the pressing of peat in thin layers most difficult, and it has been usual to separate such foreign bodies by taking the peat into suspension with water and allowing to settle. The addition of water to peat for this purpose renders preheating inefficient. It is proposed to preheat the raw peat and then to take it into suspension with water for the separation of extraneous matter, and to press the drained product. In this way the moisture content of peat can be reduced to 50%.
S. Pexton.

Combined apparatus for gas-making and coal or fuel carbonisation. A. Parker (E.P. 255,279, 7.10.25).—The plant comprises a setting of vertical retorts with an external water-gas generator. During the "blow" period the "blow" gas enters the bottom of the setting and passes in a zig-zag course along flues around the retorts, where it is burnt with air, to the stack at the top of the retort setting. During the "run" period the stack valve is closed and the "run" gas enters at the top of the setting and passes in counter-current flow through the heated flues and then up through the retort, mixing with the distillation gases. A by-pass is provided in the gas main to remove distillation gases from the retorts during the "blow" period. A. C. Monkhouse.

Coke-ovens. O. Y. Imray. From N. V. Silica en Ovenbouw Mij. (E.P. 256,444, 11.11.25).—In order to allow the heating zone in the vertical flues of a coke oven to be raised, a partition is provided between the fuel gas and secondary air ports. The partition is built up of refractory tiles which are supported in vertical slots in the flue walls, and can be introduced or withdrawn through the top of the oven battery. S. Pexton.

Coking coal. H. H. Culmer (U.S.P. 1,593,208—9, 20.7.26. Appl., [a] 19.1.20; [b] 22.1.20).—(a) The coal in the oven is separated in a number of discrete masses by non-coking fillers; during carbonisation the gases evolved penetrate the masses of coal and escape through the holes produced by the carbonisation of the fillers. (b) At the entrance and exit of the oven is a liquid seal, and bulk charges of coal are conveyed through the oven by means of a movable coal container which travels through the oven, and is diverted at the point where the coke is discharged. A. C. Monkhouse.

Carbonisation of coal. C. Hayes, Assr. to Coal Carbonisation Co. (U.S.P. 1,595,934, 10.8.26. Appl., 10.12.25).—Coal is carbonised in an externally heated cylinder which is caused to rotate through a given arc first in one direction and then in the reverse direction through a greater arc, and so on. This constant reversal of the direction of rotation prevents the material consolidating on the walls of the retort. S. Pexton.

Gas production. H. Hebel (F.P. 604,424, 10.10.25. Conv., 30.9.25).—The tar formed in the distillation of coal and separated from the gas is returned to the carbonising chamber. S. Pexton.

Distillation of bituminous material of low calorific value, such as shale. Jura Oelschiefer-Werke A.-G., and K. Nagel (G.P. 427,781, 29.11.21).—Gases circulating repeatedly through a distillation chamber containing shale or similar material, are mixed, before re-entry into the chamber, with air or oxygen in such quantity that the charge does not fuse or sinter, but a portion of the bitumen is burnt to supply the heat necessary for the distillation. L. A. Coles.

Carbonising and gasifying apparatus. R. Zeidler (G.P. 428,143, 18.3.23).—Fuel is successively distilled and carbonised on a moving grate, hot fuel gas being caused to traverse the material from above and beneath.
S. Pexton.

Retort for the continuous production of low-temperature coke. F. and Y. Zuyderhoudt (G.P. 428,208, 26.9.24).—To obtain a product similar to "coalite," coal is carbonised in a multiple-chamber circular kiln. Each chamber has self-closing doors at the top and the bottom for charging and discharging. The chambers are continuously revolved through a surrounding carbonising oven, in the sole of which is an opening communicating with a pit sufficiently deep to take the charge in bulk as it leaves the retort.
S. Pexton.

Continuous production of coke and gas. H. Koppers (G.P. 428,535, 16.1.25).—Coal is carbonised in vertical chamber ovens which have vertical heating flues on the two major faces of each oven. The two groups of heating flues each communicate with upright regenerators at the top, and at the bottom are interconnected. The heating takes place from the hot regenerator down one side of each oven and up the other, the waste gases passing to the other regenerator. Periodically the direction of gas flow is reversed.
S. Pexton.

Carbonising apparatus. O. Leissner (G.P. 428,536, 25.6.24).—Dry coal, shale, or peat is conveyed continuously through a carbonising chamber. The conveyor is operated from outside the chamber, and the inlet and outlet for the conveyor buckets are opened and closed by automatic doors which exclude air from the chamber. S. Pexton.

Low-temperature distillation of coal, shale, and wood. K. Matthaei (G.P. 430,159, 8.5.24).—The material is heated in a rotating drum enclosed in a housing containing the heating medium. The sides of the drum, which is closed at the ends, are constructed of longitudinal strips of sheet iron so arranged that the heating gases can pass between them, projections being provided on the interior to cause the contents of the

drum to be distributed freely over the surface, and to allow free contact with the hot gases. L. A. COLES.

Coking peat in the presence of tar oil of b.p. above 350°. M. DICKERT (G.P. 431,156, 29.7.20).—Peat is heated with tar oil until the distillation of oil slackens, *i.e.*, to about 330°, and, after removal of the solid residue, the oil is used again in the process. L. A. COLES.

Utilisation of material containing lime and bitumen [oil shale and oil-bearing chalk]. E. SCHWARZENAUER (G.P. 431,255, 15.9.25).—Bitumen is removed from the material, *e.g.*, by distillation, extraction, or combustion, and the residue is used for supplying the calcium in synthetic nitrogen compounds such as calcium nitrate or calcium cyanamide. The bitumen, or oils or gases derived from it, are burnt to supply the heat necessary for the production of the nitrogen compounds, or the heat so obtained is converted into electrical energy, which is utilised for the same purpose. L. A. COLES.

Distillation of coal. H. PAPE (G.P. 431,322, 3.6.23)—An ascending stream of hot gases passes through a series of superposed distillation chambers each charged with coal graded into particles of a definite size, and the initial pressure of the gases entering each chamber is regulated according to the size of the coal particles therein. L. A. COLES.

Gaseous fuels. I. G. FARBENIND. A.-G. (E.P. 249,162, 19.8.26. Conv., 16.3.25).—Producer-gas and water-gas can be greatly improved as fuels if at least 0·05% by weight of iron carbonyl is added to them. This may be done by injection of the liquid carbonyl into the gas stream or by by-passing a portion of the gas over the liquid or solid metal carbonyl or by intercalating small layers of iron or iron compounds with the coke in the producer. S. PEXTON.

Gaseous fuel [for metal cutting]. J. HARRIS (E.P. 255,250, 19.8.25).—A gas having a high ignition temperature and a low flame temperature, such as coal gas, is mixed with ether in the proportion of 1—2 lb. of ether to 200 cub. ft. of gas, *i.e.*, $2\frac{1}{2}$—5% of ether by volume. The gas is stored in cylinders under a pressure of 1800—2000 lb. per square inch, and ordinary blowpipe tips are used in the burners. A. C. MONKHOUSE.

[Oil-]gas generator. J. GIBSON (U.S.P. 1,596,248, 17.8.26. Appl., 22.6.25).—The generator comprises a hexagonal chequered chamber surmounted by spaced arches and heated by gas burners disposed in the opposite walls towards the base of the chamber. Oil and steam are admitted towards the top of the chequered portion, and oil gas is led off through outlets in the combustion chamber. S. PEXTON.

Purifying blast-furnace gases. W. and H. MATHESIUS (E.P. 255,344, 1.2.26).—The gases after a rough separation of the dust by dust pockets are subjected to electrical purification at above 40°. The remainder of the dust which originates from sublimation or chemical reaction is removed by passing the gas through two chambers into which water is sprayed in a fine state of division to produce fog. Water at 35° is used in the first chamber and cold water in the second. The drops produced are separated by centrifugal action in a Theisen washer or fan. A. C. MONKHOUSE.

Rotary gas washer, absorption apparatus, etc. Disintegrators in gas washers, absorption apparatus, etc. M. AURIG and G. BRUCKLMAYR (E.P. 257,208—9, 17.5.26).—(A) A rotating, hollow, truncated, perforated cone is supplied with liquid at its smaller end, which faces the stream of gas to be washed, and is provided on the outside with vanes, which may be straight or curved, may extend the whole length or not, and may be perforated or not. When curved they are so inclined as to throw the liquid against the approaching gas. (B) Methods of attaching cage bars of angle iron to heads of disintegrator cages, also of angle iron, are described. B. M. VENABLES.

Apparatus for treating gas liquor. LE R. W. HEFFNER and W. TIDDY, Assrs. to RAINEY-WOOD PROCESS CORP. (U.S.P. 1,595,602, 10.8.26. Appl., 22.12.25).—An ammonia still maintained at a temperature of at least 100° is provided with an additional source of ammonia to increase further the concentration of the liquor, and thus assist in the removal of phenolic substances. S. PEXTON.

Apparatus for recovering phenols from ammoniacal liquor. LE R. W. HEFFNER and W. TIDDY, Assrs. to RAINEY-WOOD PROCESS CORP. (U.S.P. 1,595,603, 10.8.26. Appl., 22.12.25).—Liquor on its way to the ammonia still is preheated in a vessel which is vented to the still, so that the distillation products from preheating pass into the still. The phenol distils over as ammonium phenoxide with the ammonia (cf. following abstract). S. PEXTON.

Treatment of gas liquor. LE R. W. HEFFNER and W. TIDDY, Assrs. to RAINEY-WOOD PROCESS CORP. (U.S.P. 1,595,604, 10.8.26. Appl., 21.1.26).—Liquor is washed with benzene which partially removes the phenols. The benzene extract is subsequently washed with caustic soda and the phenols are recovered in the usual manner. In the distillation of the liquor to recover ammonia a temperature of 98° or higher is maintained so that any remaining phenol is driven off as ammonium phenoxide with the ammonia. S. PEXTON.

Obtaining sulphur-free gases in distillation of coal. VORENBUSCH & Co. (G.P. 430,904, 29.4.24).—"Roll cinder," obtained from the rolling mills or hammers, is added to the coal. It consists of Fe_3O_4, and during gasification of the coal reacts thus :—$Fe_3O_4 + 4C = 3Fe + 4CO$; $Fe + S = FeS$. The quality of the coke is not deleteriously affected. A. DAVIDSON.

Motor fuel. A. S. NILSON (E.P. 256,131, 15.2.26).—Siphon water from coal gas distributing systems is mixed with sulphite spirit (alcohol) and heavy hydrocarbons from tar production in the proportion 4 : 1 : 2 respectively, together with caustic soda solution if desirable. W. N. HOYTE.

Preparation of fuel oil. E. A. RUDIGIER, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,597,292, 24.8.26. Appl., 21.11.24).—The viscous sludge obtained from the acid treatment of hydrocarbon oil is mixed with a

less viscous fuel oil of approximately the same specific gravity. S. Bowman.

Fuel for internal-combustion engines. E. F. Chandler (U.S.P. 1,597,343, 24.8.26. Appl., 21.11.24).—A mixture of gasoline, acetone, and water. S. Bowman.

Motor fuel. Benzol-Verband Ges. m.b.H. (Swiss P. 105,846, 13.2.23. Conv., 30.9.22).—Benzine and like substances of b.p. below 70° are mixed with heavy oils, and hydroxy-compounds such as phenols are added to increase the stability of the mixture. S. Pexton.

Separating hydrocarbons from oil-bearing earths. G. J. Rockwell (U.S.P. 1,594,796, 3.8.26. Appl., 10.4.23).—The lighter oils are first distilled off, the earth is then mixed with water and heated with steam, and agitated to free the grains from oil by attrition. The oil, water, and earth are then caused to separate in layers according to their respective specific gravities. W. N. Hoyte.

Treatment of petroleum oils. E. C. R. Marks. From Solar Refining Co. (E.P. 254,784, 3.4.25).—Skimmed (topped) petroleum, or some crude petroleum oil, is treated with alcohol, preferably anhydrous, in extraction tanks provided with baffle plates, at a temperature of 65—80°. Four extraction units are used, and in starting operations a batch is treated in the first unit till the alcohol has extracted the light portion or cracking stock, after which the oil is passed to the second unit, in which the light lubricating oils are extracted, thence to the third unit, where lubricating oil of 250—310 sec. viscosity Saybolt is extracted, and further to the fourth unit, where lubricating oil of 450—500 sec. viscosity Saybolt is extracted. The residue is an asphalt with a very low content of free carbon and with good binding qualities, flowing slowly at 38°. The alcoholic extract from each unit is allowed to settle and run to a still, where the alcohol is distilled off and dehydrated by treatment with quicklime and with anhydrous copper sulphate. It is passed to a storage tank in which bags of anhydrous copper sulphate are suspended, and then again used for extraction. The oil left in the alcohol stills is dried to remove traces of alcohol, except in the case of the cracking stock. The lubricating oils produced require once steaming and treatment to remove colour. The fraction from the third unit may undergo a further extraction to remove the less viscous constituents. The fractions of oil extracted in the respective units are 35%, 10%, 35%, and 10%, leaving 10% of residuum. The proportions of alcohol in the respective units are $1\frac{1}{2}$—2, 2—4, 3—5, and 4—6 times the amount of oil. The lubricants from a paraffin base oil have to be cold pressed to remove wax. The presence of a trace of alcohol favours chilling and pressing, after which it may be removed. The lubricants produced are of superior quality, and the yield is about $2\frac{1}{2}$ times that obtained by overhead distillation. H. Moore.

Obtaining light hydrocarbons and carbon from heavy hydrocarbons, coal tar, pitch, etc. W. Knapp, and Wandsbeker Maschinenfabr. u. Eisenbauanst. B. Fischer & C. Steiding (E.P. 257,152, 24.12.25).—The raw material, contained in a cylindrical steel tube provided with a lid, is introduced into a red-hot horizontal retort and completely carbonised therein; the issuing vapours pass through a boiler containing a suitable oil of high b.p., and the residual gases are condensed to separate light hydrocarbons, methane, etc.

Apparatus for cracking oil. E. O. Linton, Assr. to Linton Gasoline Process Co. (U.S.P. 1,592,214, 13.7.26. Appl., 17.10.22).—The apparatus comprises a heated vaporising chamber, in which are inclined plates, above which the oil is delivered, an exit pipe at the bottom of the chamber, and a heated vapour cracking retort. The retort has a shallow passage with heated walls for the vapours, and a conduit of less diameter than the retort connects the vaporising chamber and the lower part of the retort. H. Moore.

Treatment and refining of mineral oils. J. J. Allinson, Assr. to Doherty Research Co. (U.S.P. 1,592,324, 13.7.26. Appl., 4.5.20).—Crude petroleum containing natural brines is confined and heated in a rapidly moving stream by heat interchange with hot oil residuum. Brine is separated from the oil, which is heated again by heat interchange with residuum, then expanded, and vapour, additional brine, and residuum are separated. The apparatus comprises a connected series of heaters, a settling tank between successive heaters, a vapour tower connected to the last heater, a connected series of oil stills, and a salt and water separator between the tower and the stills. H. Moore.

Treatment of petroleum products. J. C. Black, W. D. Rial, and R. T. Howes (U.S.P. 1,592,329, 13.7.26. Appl., 1.6.25).—Acid-treated cracked products with boiling points similar to that of gasoline are purified by treating with weak alkali to neutralise part of the acid. The alkaline solution is removed from the products, which are then mixed with a strong alkaline solution and heated to not above 175°. Vaporisation is prevented, and the alkali breaks down gums and gum-forming substances. The mixture is then cooled, the alkali separated, and the products are mixed with a heavier hydrocarbon and distilled to remove gum and gum-forming substances. H. Moore.

Cracking [hydrocarbon] oil. P. Danckwardt (U.S.P. 1,594,666, 3.8.26. Appl., 6.10.24).—The vapours from a cracking still are passed through a mixture of molten sodium hydroxide and nickel. W. N. Hoyte.

Producing gasoline. H. M. Weir, Assr. to Standard Development Co. (U.S.P. 1,594,915, 3.8.26. Appl., 8.4.21).—Gas containing gasoline is stripped by passage through a solid absorbent; this is subsequently heated, and the vapours evolved are passed through a heated heavy petroleum distillate containing gasoline; the resulting vapours are condensed. W. N. Hoyte.

Apparatus for treating oil. G. Egloff and H. P. Benner, Assrs. to Universal Oil Products Co. (U.S.P. 1,595,179, 10.8.26. Appl., 20.8.20. Renewed 14.1.26).—The apparatus comprises a rotary still and stationary still connected by a transfer line. Means are provided for heating the upper part of each still independently, for withdrawing vapour from one of the stills, and for maintaining a pressure on the stills. H. Moore.

Apparatus for conversion of oils. J. H. Adams, Assr. to Texas Co. (U.S.P. 1,595,332, 10.8.26. Appl., 22.6.23).—Oil is passed continuously under pressure through a number of vertical tubes mounted in a furnace and heated to cracking temperature. The tubes extend through the upper and lower walls of the furnace, and are connected at their lower ends with a collecting chamber for carbon and residual oil, and at their upper ends with a vapour and liquid-separating chamber. The latter contains a number of interconnected overflow pipes immersed in oil in the chamber, in order to facilitate the separation of vapour and to maintain a constant oil level. The liberated vapours are removed and condensed.

Fractionally distilling and condensing oils. E. M. Clark and F. A. Howard, Assrs. to Standard Development Co. (U.S.P. 1,595,642, 10.8.26. Appl., 5.5.22).—Hydrocarbon vapours pass through a series of heat exchangers, into one of which water at nearly its boiling point is introduced and travels in the same direction as the vapours, being thereby converted into steam. The steam is delivered to another heat exchanger in indirect heat contact with the vapours before these pass to the first heat exchanger. Boiling water is mixed with the steam before it enters the second heat exchanger, and the mixture flows in the opposite direction to the hydrocarbon vapours in the heat exchanger.
H. Moore.

Recovery of hydrocarbon vapours and derivatives thereof. G. G. Oberfell, Assr. to Gasoline Recovery Corp. (U.S.P. 1,595,681, 10.8.26. Appl., 6.12.19).—The gasoline in natural gas is absorbed by activated carbon, and is subsequently recovered by distillation with steam. H. Moore.

Recovering condensable vapours from gas mixtures. G. G. Oberfell and G. A. Burrell, Assrs. to Gasoline Recovery Corp. (U.S.P. 1,595,682, 10.8.26. Appl., 20.5.19. Renewed 5.1.26).—Hydrocarbon vapours are absorbed from gaseous mixtures by activated charcoal. The charcoal is then heated under pressure to drive off the absorbed vapours. A portion of the vapours is condensed at a relatively low temperature, and the remaining portion is compressed and condensed.
H. Moore.

Extracting vapours from gaseous mixtures. G. A. Burrell, G. G. Oberfell, and C. J. Voress, Assrs. to Gasoline Recovery Corp. (U.S.P. 1,595,683, 10.8.26. Appl., 18.6.20).—The vapours of mineral oils of boiling points covering a range of 5·5° or more are absorbed by contacting the gaseous mixture with particles of activated carbon. A portion of the vapours is absorbed almost instantaneously, and then more of the mixture is passed, so that vapours of lower-boiling oils are re-volatilised and vapours of higher-boiling oils absorbed, the process being continued until vapours of the oils it is desired to recover are absorbed by the charcoal. A volatilising agent is then introduced into contact with the charcoal to expel the absorbed vapours. The condensed portion of the vapours is separated from the volatilising agent by gravity, and a second portion of the vapours subsequently condensed by cooling and/or pressure. H. Moore.

Topping [distillation] plant for crude oil. C. F. Hanson and P. Vandervort (U.S.P. 1,597,375, 24.8.26. Appl., 29.12.22).—The still consists of a vertical oil passage fitted with a number of convex plates which tend to divide the oil flow into two streams. Gas passages are provided above each plate. S. Bowman.

Oil coking still. J. O. Jenson (U.S.P. 1,597,469, 24.8.26. Appl., 20.1.21).—The bottom of a cylindrical still is reinforced by a number of interior longitudinal beams, which also serve to distribute heat to the contents of the still. S. Bowman.

Apparatus for cracking and distillation of petroleum. C. M. Page, Assr. to G. Fabyan (U.S.P. 1,597,476, 24.8.26. Appl., 26.8.20).—A cylindrical pressure still is provided with a number of closely-situated vertical metallic surfaces, which are electrically heated. S. Bowman.

Purification of naphtha. G. Petrov (G.P. 428,812, 11.5.26. Appl., 13.5.22).—Crude naphthas are oxidised with atmospheric oxygen and afterwards treated with 60—70% sulphuric acid, heat and pressure also being applied if necessary. The unsaturated hydrocarbons are polymerised and separate. S. Pexton.

Purification of hydrocarbon oils. Deutsche Erdöl-A.-G., Assees. of F. Schick (G.P. 429,444, 19.3.21).—Crude or partially refined hydrocarbon oils are rendered colourless and odourless, and are freed from resinous and asphaltic constituents by mixing them with phenols or their derivatives, adding solvents, with the exception of ethyl alcohol, if necessary, and subsequently removing the phenol layer. L. A. Coles.

Purification of low-boiling hydrocarbons. J. D. Riedel A.-G. (G.P. 430,974, 15.5.25).—Hydrocarbon oils of b.p. up to about 200° are dissolved in hydrogenated naphthalene, preferably tetrahydronaphthalene, and subsequently separated from the solvent by distillation. Alternatively, the hydrocarbons are passed in the form of gas or vapour through a quantity of hydrogenated naphthalene maintained above the boiling point of the hydrocarbons. L. A. Coles.

Method of dehydrating oil emulsions. R. A. Halloran, Assr. to Standard Oil Co. (U.S.P. 1,597,461, 24.8.26. Appl., 11.6.25).—The emulsion is mixed with 0·5% by volume of acid tar obtained from the treatment of kerosene with sulphuric acid, and heated to 80°.
S. Bowman.

Manufacture of lubricants. M. C. van Gundy and H. Dimmig, Assrs. to Texas Co. (U.S.P. 1,594,762, 3.8.26. Appl., 13.10.21).—A mixture of oleic acid and lead oxide is heated gradually to 270—274° and maintained at that temperature until no free acid is present; the resulting compound is mixed with the lubricating oil.
W. N. Hoyte.

Manufacture of sulphonic acids and salts thereof from mineral oils. P. I. Schestakov (E.P. 247,940 9.2.26. Conv., 17.2.25).—The oil is treated with sulphuric acid, the acid tar is separated, and the oil is heated, if necessary under pressure, to about 100° with aqueous acetic acid or one of its esters; the sulphonic acids are thereby precipitated. Alkali sulphonates may be ob-

tained by similar treatment of the tar obtained on treating the sulphonated oils with caustic soda.
W. N. Hoyte.

See also pages 856, **Filtering apparatus** (E.P. 256,124). 857, **Heating blast for producers** (E.P. 255,281). 865, **Destructive distillation of oils** (G.P. 431,516). 876, **Removing hydrogen sulphide from gases** (G.P. 431,307). 879, **Paving materials** (E.P. 256,513). 898, **Hydrocarbon gases** (U.S.P. 1,594,823).

III.—TAR AND TAR PRODUCTS.

Distillation of tar, pitch, and coal with superheated steam in a vacuum. M. Dunkel (Mitt. Schles. Kohlenforschungsinst. Kaiser Wilhelm-Ges., 1925, **2**, 201—217 ; Chem Zentr., 1926, II, 674).—Lubricating oils and paraffins which readily decompose in distillation under atmospheric pressure may be recovered by distillation of primary tar in steam at very low pressures. In the same way volatile products can be distilled from ordinary pitch, leaving a pitch residue much harder and less fusible than the original pitch. Coal and lignite, similarly, yield tars which are distinct from their ordinary tars because the application of steam and vacuum distillation materially reduces the temperature at which their primary constituents distil. S. Pexton.

Thermal decomposition of lignite tar. R. von Walther and G. Benthin (Braunkohlenarchiv, 1926, **10**, 25—38).—A technical lignite tar rich in paraffins was pyrogenically treated in a tube which could be heated uniformly at 500°, 550°, 600°, 650°, or 700° over either one-third or two-thirds of its length. When cracking over one-third of the tube, a reaction temperature 30—50° higher than when using two-thirds was required to produce similar results. In both cases, however, the quantity of unsaturated hydrocarbons produced was a maximum at 650°. The amount of phenol increases as the temperature rises from 500° to 700°. S. Pexton.

Composition and chemical constitution of lubricants and their synthesis [from tar products]. A. Spilker (Z. angew. Chem., 1926, **39**, 997—999 ; Brennstoff-Chem., 1926, **7**, 261—264 ; cf. B., 1892, 22).—Viscosities are tabulated for the liquid hydrogenation products of certain hydrocarbons of high mol. wt., obtained from the higher boiling fractions of anthracite coal-tar and pitch, and reduced by the method of Bergius. These hydrides constitute a new class of artificial lubricating oils. Their viscosity increases with rising mol. wt. of the parent hydrocarbon. Reduction of chrysene yields, as main product, Liebermann's hydride $C_{18}H_{28}$ (B., 1889, 274), which can be distilled unchanged over heated pumice if the temperature be moderated. These substances are compared with the similarly high-boiling but differently constituted lubricants from petroleum.
L. M. Clark.

Patents.

Production of stable, non-resinifying products from low-temperature tar. Zeche M. Stinnes, and A. Weindel (G.P. 430,438, 5.9.23).—Products recovered from low-temperature tar without distillation, by the process described in G.P. 420,394 (B., 1926, 432), are treated with three or four times their bulk of benzine, and, after removal of the insoluble asphaltic constituents, the benzine is distilled off. The residue is used without further treatment, or is purified by distillation under ordinary or reduced pressure. L. A. Coles.

Destructive distillation of tars and oils. E. Blümner (G.P. 431,516, 14.1.22. Addn. to 340,991).—Liquid collecting above the fused metal in the process described in the chief patent (B., 1922, 407 A) is withdrawn by a tube projecting into the pressure chamber, the relative volumes of liquid and vapour above the fused metal being regulated by altering the length of the tube within the chamber. L. A. Coles.

[Apparatus for use in the] production of hydrocarbons from phenols. F. Fischer (G.P. 431,479, 21.6.22).—The interior of metal apparatus is coated with a layer of material capable of hindering deposition of carbon ; *e.g.*, a layer of metal sulphide is formed by passing gases containing sulphur through the apparatus before use (cf. Fischer and Zerbe, B., 1923, 1168 A).
L. A. Coles.

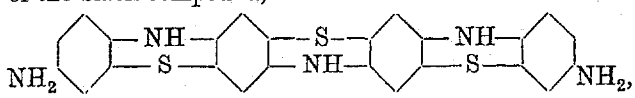
Production of material containing sulphur [for use in the manufacture of printers' ink and lubricants]. Gelsenkirchener Bergwerks-A.-G., and F. Schütz (G.P. 426,991, 20.9.22).—Material suitable for use in the manufacture of printers' ink and of lubricants having a high dropping point is obtained by heating, *e.g.*, to 200—220°, distillation products of pitch together with sulphur or with substances yielding sulphur, *e.g.*, disulphur dichloride. L. A. Coles.

Production of hard bituminous material of high fusing point from acid resins. I. G. Farbenind. A.-G., Assess. of G. Balle (G.P. 427,607, 15.3.23. Addn. to 410,012).—Acid resins or products obtained by treating them with disulphur dichloride by the process described in the chief patent (cf. B., 1925, 493) are heated with aromatic bases, such as aniline, and, after removal of excess of the base, the products are distilled at atmospheric pressure or *in vacuo*, and, if necessary, are subsequently treated with disulphur dichloride in the presence or absence of condensing agents. L. A. Coles.

See also pages 862, **Recovering phenols from gas liquor** (U.S.P. 1,595,602—4). 863, **Light hydrocarbons from tar etc.** (E.P. 257,152). 864, **Purifying hydrocarbon oils** (G.P. 429,444) ; **Purifying low-boiling hydrocarbons** (G.P. 430,974). 879, **Paving materials** (E.P. 256,513).

IV.—DYESTUFFS AND INTERMEDIATES.

Sulphur Black from benzeneazodinitrodiphenylamine. A. Beretta (Giorn. Chim. Ind. Appl., 1926, **8**, 121—122).—Fusion of 4′-benzeneazo-2:4-dinitrodiphenylamine with alkaline polysulphides at 140° results in (1) reduction to 2:4:4′-triaminodiphenylamine, (2) formation of the thiodiphenylaminic nucleus with elimination of ammonia, (3) condensation of two thiodiphenylaminic groups with further liberation of ammonia and formation of the black compound,



which dyes cotton greyish-green to black and gives

brownish-green solutions in sodium sulphide and a dark green solution in concentrated sulphuric acid. The method previously given (A., 1924, i, 221) for the preparation of the intermediate 4′-benzeneazo-2 : 4-dinitrodiphenylamine required may advantageously be replaced by the following procedure : *p*-aminoazobenzene hydrochloride (23·3 g.), 2 : 4-dinitrochlorobenzene (20 g.), anhydrous sodium carbonate (25 g.), and water (300 c.c.) are heated together over a naked flame for 24 hrs. with occasional agitation to ensure regular ebullition, the mass being filtered by suction while still hot. On cooling, the filtrate yields sodium dinitrophenoxide derived from dinitrochlorobenzene which has not reacted. The solid mass remaining on the filter is repeatedly extracted with boiling water to free it from dinitrophenoxide and aminoazobenzene, which may be recovered by fractional crystallisation. The residual reddish-brown product is almost pure 4′-benzeneazo-2:4-dinitrodiphenylamine. T. H. POPE.

PATENTS.

Manufacture of sulphur dyestuffs. SOC. OF CHEM. IND. IN BASLE (E.P. 243,739, 25.11.25. Conv., 28.11.24. Addn. to 199,360, B., 1924, 9).—In place of the benzidine used in the original patent, there is used a derivative such as diacetylbenzidine or another aromatic base such as aniline, toluidine, chloroaniline, phenylenediamine, or derivatives of these such as acetanilide, an alkylaniline, or nitroaniline. The sulphurisation may be carried out in presence of a substratum such as common salt, or of a flux such as a naphthol or phenol. The dyes obtained have great intensity and fastness. Use of the acetylated bases gives dyes of redder shades than those obtained by using the bases themselves. A. DAVIDSON.

Arylidoanthraquinone derivatives. FARBW. VORM., MEISTER, LUCIUS, & BRÜNING (E.P. 244,450, 28.11.25. Conv., 12.12.24).—The compounds are obtained in yields of over 80% by heating a 5 : 8-dihalogenoquinizarin with a primary aromatic amine in the absence of copper powder, with or without a condensing agent. 5 : 8-Dianilidoquinizarin, m.p. 258—260°, prepared by this method, differs from the product obtained by using copper powder. The corresponding 5 : 8-*di*-p-*toluidido*-derivative melts above 270°. Both yield sulphonic acids which dye wool pure, fast, greenish-yellow shades from an acid bath. J. S. H. DAVIES.

Manufacture of new azo-dyestuffs [from o-aminobenzaldehyde]. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 245,128, 21.12.25).—The unstable azo-dyes produced by coupling diazotised *o*-aminobenzaldehyde or its substituted derivatives with the usual azo-components are rendered stable by treatment with hydroxylamine. The new *dyes* yield metallic compounds, *e.g.*, with copper and chromium salts, which may be prepared in substance or formed on the fibre, and which possess improved fastness qualities. For example, 121 pts. of *o*-aminobenzaldehyde are diazotised and coupled with 337 pts. of sodium 2-phenylamino-8-naphthol-6-sulphonate in sodium carbonate solution. The product is salted out, filtered off, and washed, then suspended in water and stirred for 24 hrs. with a solution of 139 pts. of hydroxylamine hydrochloride and 272 pts. of sodium acetate crystals. The product dyes wool in yellowish-brown shades which on treatment with chromium salts become deeper, with increased fastness. A. DAVIDSON.

Manufacture of new aminotriarylmethanes and of azo dyestuffs derived from them. BRIT. DYESTUFFS CORP. and K. H. SAUNDERS (E.P. 256,775, 4.8.25).—*Aminotriarylmethanes* are obtained by condensing tetra-alkyldiaminobenzhydrol with the aminosulphones obtained by the process described in E.P. 245,865 (B., 1926, 233). The derived monoazo-dyes produce yellow to red shades on animal fibres from an acid bath. The fixation on after-chroming is accompanied by oxidation to triarylmethane dyes containing a mixed chromophore. For example, the dye obtained from tetramethyldiaminobenzhydrol by successive condensation with the aminosulphone derived by reduction of the condensation product of 5-sulphino-*o*-hydroxytoluic acid and 4-chloro-3-nitrobenzenesulphonic acid, diazotisation, and coupling with 1-phenyl-3-methyl-5-pyrazolone, dyes wool, or chromed wool, yellow, passing on after-chroming to bright yellowish-green, fast to milling. When the coupling component is β-naphthol or ethyl-β-naphthylamine, the primary colour is orange or red, passing on chroming to brown or violet, respectively. J. S. H. DAVIES.

Azo dyes. A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 256,808, 23.9.25).—Azo dyes fast to kier boiling are produced in substance or on the fibre by coupling diazotised, unsulphonated aminodiphenyl compounds, their homologues, and substitution products with arylides of 2 : 3-hydroxynaphthoic acid or bis-2 : 3-hydroxynaphthoylarylenediamines etc. The fibre impregnated with the arylide need not be dried before developing with the diazotised amine. The shades produced by a number of such azo dyes are described. 4 : 4′-*Dichloroaminodiphenyl*, m.p. 95—96°, and 4 : 4′-*dichloroamino*-3 : 3′-*ditolyl*, m.p. 88°, are obtained from 4 : 4′-dichlorodiphenyl and 4 : 4′-dichloro-3 : 3′-ditolyl, respectively, by successive nitration and reduction. J. S. H. DAVIES.

Azo dyes. A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 256,809, 23.9.25).—Azo dyes fast to kier boiling are produced in substance or on the vegetable fibre by coupling diazotised unsulphonated amino-ψ-azimines, obtained by reducing the corresponding nitro-compounds, or by the method of Schmidt and Hagenböcker (A., 1921, i, 897 ; G.P. 338,926, B., 1921, 749 A), with arylides of 2 : 3-hydroxynaphthoic acid, bis-2 : 3-hydroxynaphthoylarylenediamines, and the like. The fibre, impregnated with the arylide, need not be dried before developing with the diazotised amine. The shades produced by a number of such azo dyes are described. 2-(2′-Aminophenyl)-, 2-(3′-aminophenyl)-, 2-(4′-aminophenyl)-, 2-(3′-amino-4′-methylphenyl)-, 2-(3′-amino-4′-methoxyphenyl)-, 2-(2′-amino-4′-methylphenyl)-, 2-(2′-amino-4′-chlorophenyl)-αβ-naphtha-1:2:3-triazole melt at 120°, 160°, 203—205°, 172—173°, 185°, 137—138°, and 192°, respectively. J. S. H. DAVIES.

Azo dyes containing a diphenylurea [diphenylcarbamide] nucleus. H. WENKER, Assr. to NAT. ANILINE & CHEMICAL CO. (U.S.P. 1,594,805, 3.8.26. Appl., 6.11.25).—A mixture of 1 or 2 mols. of a *p*-aminobenzeneazoarylsulphonic or carboxylic acid, which

contains only one free amino-group and 1 mol. of a disazo dye of the general formula $p\mathrm{NH_2R''N:N(2)NH_2}$ (1) $\mathrm{C_{10}H_{4-x}(SO_3H)_xOH(8)N:NR'''Y(7)}$ where R'' is a benezene radical which may be further substituted, R''' is a benzene radical, x is 1 or 2, and Y is H, NH_2 or substituted NH_2, or a substituent other than NO_2 or OH, is treated with phosgene in presence of an acid-binding agent such as sodium carbonate. If Y in the formula is NH_2 2 mols. of the *p*-aminobenzeneazoarylsulphonic or carboxylic acid component are used, otherwise only 1 mol. is taken. The products dye cotton green shades fast to light and washing, which can readily be discharged by the action of sodium hyposulphite or the like. As an example, *p*-aminobenzeneazosalicyclic acid is treated in sodium carbonate solution at 50—60° with phosgene in presence of 1 mol. of *p*-aminobenzene-2-azo-1-amino-8-maphthol-3 : 6-disulphonic acid-7-azobenzene to yield a dye giving fast green shades on cotton from a neutral or alkaline bath. T. S. WHEELER.

Bisazo dyestuffs containing a diphenylurea [diphenylcarbamide] nucleus. L. W. GELLER, Assr. to NAT. ANILINE & CHEMICAL Co. (U.S.P. 1,594,828, 3.8.26. Appl., 28.4.24).—By the action of phosgene in presence of an aqueous solution of sodium carbonate on a mixture of 4'-amino-4-hydroxyazobenzene-5-carboxylic acid and 4'-chloro-4-amino-5-alkoxy-2-methylazobenzene-5'-sulphonic acid, dyes are obtained which give reddish-yellow to greenish-yellow shades, fast to light and washing, on unmordanted cotton capable of being discharged to a white with sodium hyposulphite. They also dye wool, silk, and other fibres. As an example, 1·3 mols. of 4'-amino-4-hydroxyazobenzene-5-carboxylic acid and 1 mol. of 4'-chloro-4-amino-5-methoxy-2-methylazobenzene-5'-sulphonic acid give with phosgene in sodium carbonate solution a dye which yields lemon yellow shades on unmordanted cotton and yellow shades on wool and silk. By varying the proportions of the two components products giving reddish-yellow and greenish-yellow shades can be obtained. T. S. WHEELER.

Preparation of diazophenolsulphonic acids and their nuclear substituted derivatives. V. M. RODIONOV, V. K. MATWEEV, and "ANILTRUST" (G.P. 430,885, 13.8.24).—Aqueous solutions of salts of phenolsulphonic acids or their nuclear substitution products are treated with sodium nitrite and a mineral acid at a low temperature. For example, from an aqueous solution of sodium phenol-*o*-sulphonate and sodium nitrite at 0°, acidified slowly with 25—30% sulphuric acid, crystals of 4-*diazophenol*-2-*sulphonic acid* are obtained; yield 80%. Similarly, from sodium phenol-*p*-sulphonate and nitrite with hydrochloric acid at 20°, a solution of 2-*diazophenol*-4-*sulphonic acid* is obtained, this compound being more soluble than its isomeride. The azo-compound formed by coupling the latter diazo-solution with β-naphthol is obtained in 50—60% yield. Treatment of 2-chlorophenol-4-sulphonic acid with nitrite and hydrochloric acid (*d* 1·1) at 0° gives a solution of 2-*chloro*-6-*diazophenol*-4-*sulphonic acid*, which coupled with β-naphthol yields an azo dye, which dyes wool and silk brownish-red, changing to garnet-red by after-chroming and to cherry-red by treatment with copper salts. A. DAVIDSON.

Diazotisation of aminophenols. RHEINISCHE KAMPFER-FABR. G.m.b.H., Assees. of S. SKRAUP and K. STEINRUCK (G.P. 431,513, 25.3.24).—Aminophenols which yield quinones on diazotisation by the usual process are diazotised by treatment with nitrites in the presence of mineral acids and salts of copper, iron, zinc, or similar metals. The diazo-solution can be used direct for coupling or for displacement of the diazo-group by halogens or other substituents. For example, amino-xylenol sulphate dissolved in dilute sulphuric acid containing zinc sulphate is diazotised at 0° with sodium nitrite, and the product, on coupling with sodium β-naphthol-3 : 4-disulphonate in sodium carbonate solution, yields a *dye* giving bluish-red shades. The diazo-solution prepared from 6-amino-*p*-*iso*propyl-*m*-cresol sulphate, on addition to copper chloride solution yields 6-chloro-*p*-*iso*propyl-*m*-cresol (chlorothymol), or on addition to a solution of stannous chloride in concentrated hydrochloric acid yields a red oil, apparently 6-hydrazino-*p*-*iso*propyl-*m*-cresol, which on boiling with copper sulphate solution splits off nitrogen, with the formation of thymol. L. A. COLES.

Manufacture of benzanthrone derivatives containing sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 255,731, 13.1.26).—Benzanthronesulphinic acids are prepared from 3-halogenobenzanthrones by the action of sodium hyposulphite (or sodium formaldehyde-sulphoxylate) in neutral or alkaline solution, using preferably a solvent which will dissolve both inorganic and organic materials. For example, 10 pts. of 3-chlorobenzanthrone are stirred at 35—40° with 200 pts. of methyl alcohol, 70 pts. of 20% aqueous ammonia, and 10 pts. of sodium hyposulphite, until the product is water-soluble. The alcohol is distilled off, the residue dissolved in hot water and, after oxidising unchanged hyposulphite with air, the solution is acidified, when *benzanthrone*-3-*sulphinic acid*, m.p. 225°, is precipitated as a brownish-yellow powder. The sulphinic acids can be oxidised to sulphonic acids, and are reduced by sodium polysulphide to mercaptans, or by sodium sulphide at 70—80° to 3 : 3'-benzanthronyl sulphides or at higher temperatures to *iso*dibenzanthrones. A. DAVIDSON.

Manufacture of intermediates [mercaptans, sulphides, and disulphides] of the benzanthrone series. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABRIK and FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 256,059, 14.8.25).—Benzanthrone mercaptans and other sulphur derivatives are obtained by the action of alkali sulphides on halogenated benzanthrones or of sulphur on nitrobenzanthrones or nitrohalogenobenzanthrones. Aryl mercaptans or benzanthrone mercaptans may also be used. Methylbenzanthrones are excluded. The reaction may be carried out in the presence or absence of diluents. For example, 10 pts. of bromobenzanthrone are boiled for several hours under reflux with a solution of 100 pts. of sodium sulphide crystals in 50 pts. of water and 100 pts. of 96% alcohol. A violet solution of the sodium salt of benzanthrone mercaptan is formed, from which the mercaptan is obtained as a yellow powder. From 3-chlorobenzanthrone and thio-*p*-cresol is obtained *benzanthrone* 3-*thio*-p-*tolyl ether*, yellow, m.p.

218—220°. Similarly, from 4-chlorobenzanthrone is obtained *benzanthrone 4-thio-p-tolyl ether*, m.p. 170—171°. 3-Nitrobenzanthrone in trichlorobenzene when heated with sulphur yields *benzanthronyl* 3 : 3′-*sulphide*, m.p. above 300°, and benzanthrone 3-mercaptan, which on passing a current of air through the solution of its sodium salt, yields benzanthronyl 3 : 3′-disulphide, yellow, m.p. 263—265°. The disulphide is converted into the sulphide by heating with copper, phenol, arylamines, or other substances capable of taking up sulphur.

A. DAVIDSON.

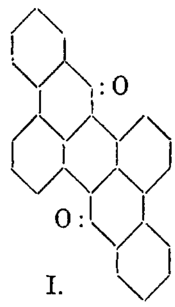
Dyestuffs and dyestuff intermediates [halogenoalkoxybenzanthrones and *iso*dibenzanthrones]. R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 256,281, 2.2.25; cf. E.P. 193,431).—A Bz-chlorobenzanthrone, m.p. 174—178°, is nitrated in nitrobenzene solution to *chloronitrobenzanthrone*, m.p. 287—290°. This is reduced in pyridine or aniline by zinc dust and hydrochloric acid, or in concentrated sulphuric acid by aluminium powder, to the *chloroaminobenzanthrone*, the product when aluminium is used being red crystals, m.p. 280—281°. The amino-compound, diazotised in the usual way and the suspension of diazo compound boiled, yields *chlorohydroxybenzanthrone*. The latter is methylated, in suspension in nitrobenzene, with methyl sulphate to *chloromethoxybenzanthrone*. Alcoholic potash at 110° converts this into what appears to be *dimethoxy*iso*dibenzanthrone*, which dyes cotton a reddish-blue of great fastness. A. DAVIDSON.

Reduction of vat dyes to leuco-compounds. COMP. NAT. DE MATIÈRES COLORANTES ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES, ETABL. KUHLMANN (F.P. 601,632, 6.11.24).—A suspension of the dye in a dry organic solvent is treated with hydrogen in presence of catalysts, such as nickel, cobalt, iron, copper, either alone or mixed with their lower oxides. Thus, a suspension of indigo in dimethylaniline, containing nickel precipitated on kiesulguhr, is treated with hydrogen at 90—100°. The resulting suspension of leuco-indigo is suitable for conversion into esters. J. S. H. DAVIES.

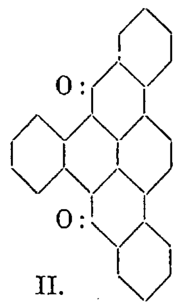
Preparation of halogenated dibenzpyrenequinones. I. G. FARBENIND. A.-G., Assees. of G. KRÄNZLEIN, R. SEDLMAYR, and H. VOLLMANN (G.P. 430,556, 11.6.24).—Dibenzpyrenequinones are treated with halogens or halogenating agents in presence or absence of carriers. The halogenodibenzpyrenequinones so obtained are partly themselves vat dyes, and partly intermediates for dibenzpyrenequinone dyes. By chlorination of dibenzpyrenequinone in sulphuric acid monohydrate, with addition of iodine, a *chlorodibenzpyrenequinone*, brownish-yellow, m.p. above 350°, is formed.

A. DAVIDSON.

Manufacture of vat dyes of the anthracene series. I. G. FARBENIND. A.-G., Assees. of A. WOLFRAM (G.P. 430,557, 11.6.24).—9 : 10-Dibenzoylanthracenes, or their derivatives and substitution products, are heated with metal chlorides at high temperatures in presence of air or oxygen and of an organic flux such as benzophenone, diphenylsulphone, etc. The dyes formed, which possess a structure of the types I or II,



I.



II.

dye cotton from the vat in red-violet to blue shades.

A. DAVIDSON.

Preparation of nitrogenous condensation products of the anthraquinone series [anthraquinonylcarbazoles]. I. G. FARBENIND. A.-G., Assees. of W. MIEG (G.P. 430,884, 13.1.22).—Chloro- or bromoanthraquinones or their derivatives are heated with carbazole, with or without a diluent, in presence of an acid-binding agent and of a little copper or copper salt. With bromoanthraquinones sodium acetate generally suffices as acid-binding agent, but with chloroanthraquinones stronger bases are necessary, such as calcium hydroxide or potassium carbonate. The products are *N*-substituted anthraquinonylcarbazoles, very reactive substances of intense colour, which serve as intermediates for dyes. For example, 1-chloroanthraquinone, heated to the boil for 8 hrs. with carbazole in nitrobenzene, with addition of calcium hydroxide and a little copper acetate, yields N-1-*anthraquinonylcarbazole*, reddish-orange, m.p. 260°. Similarly, from 1 : 5-dichloroanthraquinone and carbazole is obtained an orange-red condensation product in which both chlorine atoms are replaced by carbazole residues. The condensation *product* from 1 : 9-pyridone-4-bromoanthraquinone and carbazole is brownish-orange. From 2-bromoanthraquinone and carbazole in nitrobenzene, with addition of potassium carbonate and copper powder, is obtained N-2-*anthraquinonylcarbazole*, orange prisms. It gives an emerald-green solution in sulphuric acid, changing after a short time at 15° to blue-black, with formation of a *sulphonic acid* which dyes wool in clear yellow shades. A. DAVIDSON.

Manufacture of mordant dyes of the phenanthraquinone series. K. BRASS (G.P. 430,631, 25.1.23).—Aminophenanthraquinones are converted through their diazonium perbromides into phenanthraquinone azides, and these are decomposed by 65—70% sulphuric acid. 2-Aminophenanthraquinone yields, in concentrated sulphuric acid with sodium nitrite, the 2-diazonium sulphate, which in aqueous solution with bromine forms the 2-diazonium perbromide, an oily mass solidifying after some time. With 25% ammonia the aqueous suspension of the perbromide gives *phenanthraquinone 2-azide*, cinnabar-red or coppery leaflets, m.p. 186°. On warming this with 65—70% sulphuric acid there is formed (besides some 2-aminophenanthraquinone which can be extracted with alcohol) 2-*amino-3-hydroxyphenanthraquinone*, which dyes unmordanted wool greenish and chrome-mordanted wool olive-green. From 4-aminoanthraquinone is obtained similarly *phenanthraquinone 4-azide* (garnet-red, explodes on heating), which on warming with 65% sulphuric acid and diluting with an equal volume of

water yields 4-*amino*-1-*hydroxyphenanthraquinone*, violet-black needles, decomp. 217°, which dyes chrome- or alumina-mordanted wool blue-grey. If the 4-azide, after treatment with 65% sulphuric acid, is diluted with the fourfold volume of water, 1 : 4-dihydroxyphenanthraquinone, brown-violet (*diacetyl* derivative, golden-yellow, m.p. 183°) is formed. It dyes wool violet-black. Its *quinoxaline* is reddish-yellow, m.p. 288°.

A. DAVIDSON.

Manufacture of grey to black vat dyes. I. G. FARBENIND. A.-G., Assees. of P. NAWIASKY and E. KRAUCH (G.P. 430,704, 18.9.24. Addn. to 411,693; cf. also G.P. 423,878).—In place of the nitration products of dibenzanthrone, as described in the preceding patents (cf. B., 1926, 577), dibenzanthrone itself is treated with sulphur-yielding materials, especially in presence of carriers such as copper, antimony, or phosphorus compounds.

A. DAVIDSON.

Manufacture of chlorohydroxyanthraquinone. H. DODD, W. C. SPRENT, and UNITED ALKALI CO., LTD. (E.P. 256,068, 27.8.25).—Chlorohydroxyanthraquinone free from hydroxyanthraquinone is prepared by the condensation of phthalic anhydride with *o*- or *p*-chlorophenol in sulphuric acid in the absence of boric acid or aluminium chloride, so as to avoid replacement of chlorine by hydroxyl. For example, 225 g. of phthalic anhydride are dissolved in a mixture of 670 c.c. of sulphuric acid (*d* 1·84) and 80 c.c. of 20% oleum. Pure *o*-chlorophenol (193 g.) is then run in with stirring and the mixture heated at 150—160° for 5 hrs., then at 180—190° for 3 hrs., and finally at 195—200° for 3—4 hrs. The cooled melt is run into 4 litres of water at 40° and the precipitated chlorohydroxyanthraquinone, orange, m.p. 242°, is filtered off.

A. DAVIDSON.

Making 2-hydroxynaphthalene-6-carboxylic acid. F. ANDRE, Assr. to GRASSELLI DYESTUFFS CORP. (U.S.P. 1,593,816, 27.7.26. Appl., 12.11.25. Conv., 16.8.24).—182 pts. of dry potassium β-naphthoxide are heated with carbon dioxide under pressure in an autoclave for 8 hrs. at 170—230°, when 2-*naphthol*-6-*carboxylic acid*, m.p. 245° (68 pts.), and 2-naphthol-3-carboxylic acid are formed.

T. S. WHEELER.

Flaked β-naphthol. N. E. VAN STONE and C. E. DEEDS, Assrs. to SHERWIN-WILLIAMS CO. (U.S.P. 1,594,390, 3.8.26. Appl., 13.2.24).—A revolving drum partially immersed in molten β-naphthol takes up a thin film of the material, which, after solidification, is removed by a scraper in the form of thin flakes of uniform thickness. These are easily handled, are non-dusting and non-caking, and on storage do not darken as rapidly as the powder form.

T. S. WHEELER.

Production of α-nitronaphthalene-β-sulphonic acids. R. A. NELSON, Assr. to NATIONAL ANILINE & CHEMICAL CO. (U.S.P. 1,594,547, 3.8.26. Appl., 3.5.20).—500 pts. of naphthalene are treated at 160° during 15 min. with 610 pts. of sulphuric acid (100%), and a further 310 pts. of 100% sulphuric acid are then added to the sulphonated product at 75°. The product is treated with 330 pts. of nitric acid (67·5%) at 45—60° with cooling during 6—10 hrs., after which it is diluted with water and neutralised with lime. The product consists of a mixture of 1-nitronaphthalene-6- and -7-sulphonic acids together with a small amount of the 1 : 3-acid. If desired, it may be reduced without isolation to give the corresponding amino-acids (Cleve's acids).

T. S. WHEELER.

Preparation of chloro-derivatives of β-naphthol with free 1-position. I. G. FARBENIND. A.-G. (Assees. of W. HERZBERG, O. SPENGLER, and A. SCHMID (G.P. 431,165, 31.10.23).—Polychloro-derivatives of β-naphthol containing chlorine in the 1-position are treated with reducing agents; *e.g.*, 1 : 3-dichloro-2-hydroxynaphthalene dissolved in alcohol is boiled with coppered zinc dust for 15 hrs. under reflux. The hot filtered solution is diluted with water and the precipitate crystallised from benzine with addition of animal charcoal, yielding 3-*chloro*-2-*hydroxynaphthalene*, m.p. 93°. Similarly, from 1 : 3 : 4-trichloro-2-hydroxynaphthalene is obtained a mixture in equal proportions of 3-chloro-2-hydroxynaphthalene and 3 : 4-*dichloro*-2-*hydroxynaphthalene*, m.p. 108°, separable by crystallisation. The latter can also be obtained from 1 : 3 : 4-trichloro-2-hydroxynaphthalene by reduction with ferrous sulphate and the calculated quantity of sodium hydroxide. Reduction of 1 : 6-dichloro-2-hydroxynaphthalene by ferrous sulphate and sodium hydroxide yields 6-*chloro*-2-*hydroxynaphthalene*, m.p. 115°. The chloro-compounds may be used as intermediates for dyes and medicinal products.

A. DAVIDSON.

Manufacture of perylene. C. H. MARSCHALK (U.S.P. 1,593,982, 27.7.26. Appl., 13.12.23. Conv., 21.12.22).—A mixture of β-dinaphthol (6 pts.), zinc dust (10 pts.), fused zinc chloride (9 pts.), and water (3 pts.) is distilled, and the orange crystals which condense from the evolved vapours are washed with dilute caustic soda solution and recrystallised from toluene; the final product has m.p. 265°.

E. S. KREIS.

Preparation of *o*-benzoylbenzoic acid. E. C. KLIPSTEIN & SONS CO., Assees. of H. G. STONE and B. H. JACOBSON (Can. P. 254,834, 28.1.24).—Benzene is added to a well-stirred mixture of phthalic anhydride and anhydrous aluminium chloride. The excess benzene is distilled and the double compound of aluminium chloride and *o*-benzoylbenzoic acid is obtained in a finely divided condition, suitable for the preparation of anthraquinone.

J. S. H. DAVIES.

Preparation of a nitro-derivative of glycosine (di-iminazolyl). K. LEHMSTEDT (G.P. 427,954, 3.8.21).—Glycosine is nitrated by heating it with fuming nitric acid and sulphuric acid at 165° until the evolution of nitrous gases ceases. Sodium nitrate, or a mixture of nitrate and nitrite, may be used in place of nitric acid. The product is precipitated on diluting the reaction mixture with water. *Tetranitroglycosine*, $C_6H_6O_{10}N_8$ or $C_6H_2O_8N_8,2H_2O$, yellow, m.p. 276° (decomp.), acts as a dibasic acid. Its red alkali salts detonate violently on heating. Reduction of the nitro-compound yields various dyes (cf. G.P. 414,962, B., 1926, 7). Boiling with ferrous chloride and hydrochloric acid splits off two of the nitro-groups as nitric oxide. Presumably the compound is 4 : 4′ : *NN′*-tetranitro-2 : 2′-di-iminazolyl.

A. DAVIDSON.

Catalytic preparation of primary aromatic amines. I. G. FARBENIND. A.-G., Assees. of O. SCHMIDT and A. JOHANNSEN (G.P. 429,102, 29.8.24).—Nitro-compounds are reduced, in the presence of a catalyst, by gases containing carbon monoxide, but kept free from metal carbonyls, particularly iron carbonyl, by excluding iron from the inner surface of the contact chamber and from the inlet pipe, by coppering these or otherwise. For example, over a contact mass at 180—250° prepared as described in Example 2 of G.P. 352,439 (B., 1922, 689 A), is led a mixture of nitrobenzene vapour and water-gas, the latter having been desulphurised in the usual way and then freed from metal carbonyls by leading over activated charcoal. The contact mass remains active for a long time. The yield of aniline is quantitative. Water and carbon dioxide are also formed, and after absorption of the latter, the gases leaving the contact chamber can be used again. The moist aniline, after distillation, is obtained quite pure. Nitro-compounds can be reduced in gaseous condition or as liquids in solution, with or without application of pressure.
A. DAVIDSON.

Preparation of 4-halogeno-1 : 8-naphthasultones. GES. F. CHEM. IND. IN BASEL (G.P. 430,551, 12.4.25. Conv., 21.5.24).—1 : 8-Naphthasultones are treated with halogens or halogen-yielding materials in presence of a suitable carrier. For example, chlorine is led into molten 1 : 8-naphthasultone containing iron powder at 160°, and the temperature is gradually raised to 200°. When the calculated quantity of chlorine has been taken up, the mass is extracted hot with tetrachloroethane, and unchanged naphthasultone removed by digestion with 5% oleum. On cooling, *4-chloro-1 : 8-naphthasultone* crystallises out. The same substance can also be obtained by stirring 1 : 8-naphthasultone with hydrochloric acid and 85% manganese dioxide while slowly heating to 80—90° until decolorisation takes place. Again, if a 65% aqueous paste of naphthasultone is treated slowly with bromine in the cold, bromination takes place immediately and *4-bromo 1 : 8-naphthasultone*, pale yellow, m.p. 140°, is formed.
A. DAVIDSON.

Preparation of oxindole-3-acetic acid. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of W. SCHOELLER and K. SCHMIDT (G.P. 431,510, 18.2.25).—Isatin and malonic acid are fused together, *e.g.*, at 135°, until no more carbon dioxide is evolved, yielding oxindole-3-methinecarboxylic acid, which on reduction with aluminium amalgam in the presence of aqueous alkali solutions, yields oxindole-3-acetic acid, m.p. 218°.
L. A. COLES.

Colour lakes (AUSTR. P. 103,470).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Sulphur content of wool. I. Inherent variations according to the type of wool. J. BARRITT and A. T. KING (J. Text. Inst., 1926, 17, T 386—395).—For the critical work described, the Carius method was preferred above others for estimating sulphur. Another important item was accurate knowledge of the moisture content, and a special weighing bottle is shown in which about 1 g. of wool could be heated in a current of dry air. Using the refinements described, it is established that the sulphur content of wool varies from about 3·03% (coarse mohair) to about 4·13% (a Welsh mountain sheep). In general, the coarse wools come at the lower end of the scale, and kempy wool is poorer in sulphur than non-kempy, even from the same sheep. The sulphur content varies in different parts of the fleece and in different shearings from the same sheep, as well as between breed and breed. J. C. WITHERS.

[Use of] wetting out agents in carbonising wool. P. KRAIS (Textilber., 1926, 7, 757).—The degree of assistance rendered to the wetting-out of wool with 4% sulphuric acid at 20° by addition of 0·5—2% of Flerhenol PF, Neomerpin, or Leonil (cf. Auerbach, B., 1926, 705) decreases rapidly in the order named.
A. J. HALL.

Microscopical examination of damaged cotton hairs by the Congo-red test and the swelling test of Fleming and Thaysen. T. B. BRIGHT (J. Text. Inst., 1926, 17, T 396—404).—The cuticle of a cotton hair stains with Congo-red to a lighter shade than the cellulose encased by it. Consequently damage to the cuticle which exposes the cellulose can be detected by observation of the stained hair under the microscope. The cotton is wetted out, swollen in 11% caustic soda, rinsed, stained in Congo-red (2% solution), and mounted in 18% caustic soda. The final swelling usually causes the cuticle to crack spirally if the hair is sound, so that unstained cellulose shows up underneath a faint pink band of cuticle. With a hair damaged by mechanical means the cellulose will already have been exposed and will have become deeply stained, presenting the appearance of a bruise. Damage by heat is shown by the appearance of spiral bands which become very numerous as the heating is prolonged, whilst badly damaged hairs fail to swell properly in the final caustic soda solution. Mildew damage often results in deep staining throughout, but actual rupture of the cuticle may not be observed. Cotton hairs damaged by sulphuric acid, however, do not give a characteristic appearance on staining, but if the damage is severe it will be observed that the hairs do not swell to the rod shape in the caustic soda.

A test developed by Fleming and Thaysen for the detection of damage by micro-organisms (B., 1920, 263 A; 1921, 764 A) has been examined on the same material (Sakel cotton) as tested by the Congo-red method. The conclusion is drawn that the two tests vary in efficiency according to the material. For detecting small amounts of mechanical damage such as might occur to cotton in spinning processes the Congo-red test appears to be the more promising. J. C. WITHERS.

Cellulose esters of higher fatty acids. I. Ester formation from cellulose and the properties of the esters. II. Ester formation from alkali-cellulose and fatty acid chlorides, and composition of alkali-cellulose. G. KITA, T. MAZUMA, J. SAKRADA, and T. NAKASHIMA (Kunststoffe, 1926, 16, 41—43, 69—70; Chem. Zentr., 1926, II, 1266).—In the presence of pyridine, cellulose combines with stearic and palmitic anhydrides at the ordinary pressure (cf. Gault and Ehrmann, B., 1923, 826 A), but the esters contain only a very small proportion of the fatty acid; esters soluble in benzene and ether may be prepared (cf. Grün and

Wittka, B., 1922, 94 A). Dried alkali-cellulose does not combine with acid chlorides in ethereal solution, even if water is previously added. When the acid chloride is in excess, the amount which combines depends on the concentration of alkali used in preparing the alkali-cellulose, and is not affected by any excess of alkali that may be present, or by the amount of acid chloride.

A. GEAKE.

[Wood] boiling diagram for sulphite liquors containing magnesium. K. BERNDT (Papier-Fabr., 1926, **24**, 561—565, 584—587).—During the wood-boiling process the sulphur dioxide content of the liquor falls regularly and continuously, independently of whether calcium or magnesium bisulphite is used; the fall is less rapid when the magnesium bisulphite liquor contains excess of base. During boiling with calcium bisulphite the concentration of active base falls rapidly during the initial stages, probably due to precipitation as neutral calcium sulphite and as the calcium salt of a lignin–sulphur compound. During the middle part of the boil the concentration remains approximately constant, but falls rapidly during the last few hours. The total fall in concentration depends on the composition of the liquor, the proportion of wood, and the procedure. When the liquor contains both calcium and magnesium the concentration of calcium follows a similar course, but that of magnesium falls only slightly during the first part of the boil and later rises again; this is ascribed to neutral magnesium sulphite and magnesium–lignin compounds being more soluble than the corresponding calcium compounds. The absence of precipitation also leads to whiter and more easily bleached pulp. When the concentration of magnesia is increased, the duration of the boil and the consumption of sulphur dioxide are also increased, but the yield of pulp is higher. The consumption of sulphur dioxide is diminished when calcium is replaced by magnesium.

A GEAKE.

Bleaching of sulphite-cellulose. L. RYS, EICHMANN & Co. (Papier-Fabr., 1926, **24**, 529—533).—In bleaching, a portion of the hypochlorite is used in chlorinating organic substances. The proportion thus used is increased by increasing acidity of the liquor, by increasing concentration and lignin content of the cellulose, and, slightly, by the addition of neutral chlorides. Under given conditions, the ratio of the chlorine used for chlorination to that used for oxidation is fairly constant during the bleach.

A. GEAKE.

Metallographic study of corrosion in the cellulose and paper industries. V. LINDT (Papier-Fabr., 1926, **24**, 513—515, 534—539).—A lecture illustrated with photomicrographs dealing with the application of metallography to the study of corrosion and to the choice of suitable metals or alloys for various purposes in the cellulose and paper industries.

A. GEAKE.

Distillation of cellulose, lignin etc. FISCHER and TROPSCH. **Production of coal from cellulose and lignin.** TROPSCH and VON PHILIPPOVICH.—See II.

Rubbered raincoats. ESCH.—See XIV.

PATENTS.

Protecting woollen goods and similar material from attack by moth. J. BLANCKE (G.P. 430,186, 18.4.23).—The material is treated successively with a tannin solution and a solution containing an antimony salt, such as tartar emetic.

L. A. COLES.

Recovery of mercury in carroting hair. RHENANIA VEREIN CHEM. FABR. A.-G., Assees. of K. PULVERMÜLLER (G.P. 431,388, 22.11.24).—The mercury-containing gases from the carroting chamber are washed with suitable liquids, treated with substances of large superficial area, or with metals capable of yielding amalgams, or treated by a combination of the above processes. For example, the gases are passed through a filter of activated silica.

J. S. H. DAVIES.

Process of treating plant material. Process and apparatus for making pulp. F. K. FISH, JUN. (E.P. 244,788—9, 16.12.25. Conv., [A] 20 and [B] 22.12.24).—(A) Cellulosic material is boiled for about 45 min. at 116° with a solution of an alkali, containing substances such as terpenes and other oils, previously extracted from similar material. Penetration is improved by evacuating the vessel containing the material and allowing the oils to distil into it from another heated vessel, before adding the alkaline solution. The liquor is continually re-used and is not exposed to the atmosphere. (B) The material obtained as above is boiled for 4—7 hours at 170° with sodium carbonate, or sulphate or other cooking liquor. This liquor is also continually re-used after strengthening, and is not exposed to the atmosphere.

A. GEAKE.

Manufacture of artifical products from viscose. BRIT. ENKA ARTIFICIAL SILK CO., LTD., Assees. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 248,750, 2.3.26. Conv., 5.3.25).—When a salt of nickel or cobalt is added to the precipitating bath for viscose, the product obtained dyes more evenly, and its affinity for dyes is increased. A suitable bath contains 10 pts. of sulphuric acid, 8 pts. of sodium sulphate, 16 pts. of magnesium sulphate, and 1 pt. of nickel sulphate to 100 pts. by weight of the bath.

A. GEAKE.

Treating fibrous vegetable materials for the production of cellulose. CHEM. FABR. GRIESHEIM-ELEKTRON, and H. WENZL (E.P. 256,757, 7.7.25).—The material is given a preliminary boil with a solution of alkali sulphites and bisulphites, *e.g.*, for 10 hrs. at 145—150°, followed by a treatment with gaseous or aqueous chlorine; both treatments are acidic in character. The liquor from the first process may be used to neutralise the hydrochloric acid produced in the second process, and to dissolve the chlorinated incrustations, and it may be utilised finally to produce alcohol or tanning materials.

A. GEAKE.

Preventing rancidity in oils used in plastic compositions (E.P. 256,654).—See XII.

Alcohol from sulphite-cellulose waste liquor (G.P. 430,076).—See XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching of wool with sulphur dioxide. Summary of the literature. E. F. H. COOK (J. Text. Inst., 1926, **17**, T 371—378).

Bleaching of wool with sulphur dioxide and sulphurous acid, and presence of a carbonyl group

in wool. J. L. RAYNES (J. Text. Inst., 1926, 17, T 379—385).—When clean, dry wool (containing less than 2% of moisture) is left in an atmosphere of sulphur dioxide it becomes lemon-yellow in colour and about 55 c.c. of gas are fixed by 1 g. of wool at 17° and 760 mm. The coloured compound is decomposed by placing the wool in a vacuum or a current of dry nitrogen, hydrogen, etc., but the final product is not "bleached" wool. The same enhanced colour is observed with sufficiently concentrated solutions of sulphurous acid, but after removing the excess of acid the wet wool is white. It appears, then, that there are at least two components of wool which combine with sulphur dioxide, one which is itself colourless but gives a labile, coloured compound, and one which is originally coloured, reacts only when sufficient water is present, and is thus bleached. No certain evidence of the reduction of a pigment with formation of sulphuric acid was obtained, and the coloured component of wool is assumed to be a carbonyl compound, reacting to give $:C(OH)\cdot O\cdot SO_2H$. Support for this view was obtained from an observation that the increment in the nitrogen content of wool after treatment with semicarbazide was three times that realised with hydroxylamine. J. C. WITHERS.

Fastness to rubbing and washing of Naphthol AS dyes. W. ROIGER (Z. ges. Text. Ind., 1925, 654; Textilber., 1926, 7, 787).—The fastness to rubbing and washing shown of Naphthol AS dyes (cf. Kielbasinski, B., 1926, 705) is improved most satisfactorily by completely removing excess of the naphthol prepare solution from the surface of the textile material before coupling with the diazotised base, but improvement may also be effected by the addition of 2—4% of Verapol or 0·2—0·4% (calc. on the weight of solution) of Cycloran to the soap and soda solution used for soaping the dyed material. The addition of tannic acid has a similar effect but darkens the resulting shade. A. J. HALL.

Indigosol O [in dyeing and printing]. G. FRIEDLÄNDER (Textilber., 1926, 7, 781—783).—Further details of large-scale processes (cf. B., 1926, 705) are described. For dyeing cotton fabrics by slop padding methods, Indigosol O may be used together with chrome dyes, such as Chrome Fast Yellow RD, Alizarin Fast Yellow GG pdr., Chrome Violet CB, Chrome Azurin DN, and Gallophenin D, the padding liquors being stable for at least 24 hrs. A suitable padding liquor contains 100 g. of Indigosol O, 10 g. of a chrome dye, 1 g. of borax, 50 g. of gum tragacanth, 90 g. of a 10% solution of sodium chlorate, 25 g. of ammonium thiocyanite, 80 g. of a 1% solution of ammonium vanadate, 35 g. of chromium acetate of 20° B. (d 1·16), and 609 g. of water, the padded fabric being afterwards dried, then steamed for 20—30 min., washed, soaped for 1 min. at 60°, and dried. The blue shade obtained by means of Indigosol O and Chrome Violet CB may be discharged to a pure white, thereby allowing the production of the imitation marine blue effects which are usually obtained by means of tannic acid discharge styles. Coloured resists are obtained by printing cotton fabric with a resist paste containing vat, chrome, basic, or ice colours, before or after impregnation (by slop padding, or by "padding direct through the nip" for fine patterns) with a solution containing Indigosol O and a mixture of substances (*e.g.*, sodium chlorate, ammonium thiocyanate, and ammonium vanadate) capable of developing the indigo shade during the subsequent steaming after drying. Alternatively, fabric is printed with a resist paste, before or after impregnation with a solution containing Indigosol O and sodium nitrite, and then developed by "padding direct through the nip" with a solution of sulphuric acid. Sodium acetate, thiosulphate, sulphite, and hyposulphite are satisfactory resist agents. Russian green effects may be obtained by developing Indigosol O with a nitrite on fabric prepared with β-naphthol. A. J. HALL.

"Immunisation" of vegetable fibres and their use in dyed and printed materials. G. TAGLIANI (Textilber., 1926, 7, 765—770).—Cotton immunised by means of *p*-toluenesulphochloride (cf. E.P. 246,609; B., 1926, 270) has a harsher and fuller handle than normal cotton, the individual fibres contracting in length and swelling with almost complete elimination of the lumen. Immunised cotton has a resistance to water about equal to that of cotton waterproofed by means of aluminium acetate and soap, and is less hygroscopic than normal cotton. Immunised cotton has a strong affinity (proportional to the degree of immunisation) for basic dyes, and its dyeing properties are similar to those of oxycellulose, and cellulose acetates and benzoates. Immunisation causes an increase of weight of the fibres of 10—12%. Dyed yarn has increased fastness after immunisation, probably due to a contraction of the active surface of the fibres. Cotton immunised by means of *p*-toluenesulphochloride is more resistant to alkalis than the cellulose esters of aliphatic fatty acids. Immunised cotton materials are resistant to the usual processes for preparing cotton for dyeing and printing, although plate-singeing and other dry treatments at temperatures exceeding 120—125° should be avoided since they produce partial decomposition. Alkalis produce swelling and saponification, but rapid processes of mercerisation or "philanising" have no harmful effect. Local immunisation of cotton fabrics may be effected (cf. E.P. 233,704; B., 1925, 956), and the resulting fabric then dyed in two colours. Immunisation does not proceed uniformly within vegetable fibres, since the esterified portions may be extracted with suitable solvents and the insoluble residue shown to have an affinity for direct dyes. There is a possibility of immunising cellulose silks during spinning and coagulation. A. J. HALL.

PATENTS.

Bleaching vegetable fibres. CHEM. FABR. MILCH A.-G., and K. LINDNER (E.P. 246,155, 14.1.26. Conv., 14.1.25).—When vegetable fibres are bleached without a previous kier boil, wetting is improved by the addition of aromatic sulphonic acids containing aliphatic side chains, *e.g.*, the palmitobenzenesulphonic acids or *iso*propylnaphthalenesulphonic acids. These are colloidal and form soluble calcium salts. Their action is improved by the addition of soluble alcohols, and they may also be added advantageously to the liquor in kier boiling. A. GEAKE.

Bleaching of wool, silk, jute, etc. with sulphur

dioxide. F. L. POLLAIN (F.P. 603,253, 28.5.25).—Bleaching is effected in a closed circular chamber. The gas mixture, containing about 10% of sulphur dioxide, generated by burning sulphur, is introduced into the upper portion, and withdrawn at the lower. Efficient ventilation is required to ensure uniform distribution of the gas. J. S. H. DAVIES.

Bleaching and removing size from raw silk fabrics (Bourrette silk). R. M. KIEN (F.P. 604,600, 20.7.25).—The silk is steeped in a solution in lime-free water of 50—60% (calculated on the weight of the silk) of sodium perborate containing 10% of active oxygen, and the temperature is raised to 70—75° during 3 hrs., using a heating coil of nickel or aluminium, after which the silk is washed successively with lime-free water and dilute acid. L. A. COLES.

Mercerising process. CHEM. FABR. MILCH A.-G., and K. LINDNER (G.P. 430,085, 28.7.25).—The duration of the mercerising process is reduced by adding to the alkali solution alcohols or ketones of high molecular weight, such as butyl alcohol, amyl alcohol, or *cyclo*hexanol, these being rendered soluble by the addition of alcohols or ketones of low molecular weight, such as methyl or ethyl alcohol. L. A. COLES.

Dyeing apparatus. F. KIRCHHOF (E.P. 245,157, 24.12.25. Conv., 26.12.24).—Greater uniformity in the dyeing of textile materials wound on bobbins is secured by mounting these on a vertical cylinder, which can be rotated in either direction in the dye liquor; the liquor is also pumped through the bobbins. A. GEAKE.

Fixation of basic dyes. I. G. FARBENIND A.-G., Assees. of K. DAIMLER (G.P. 430,679, 26.9.24. Addn. to 399,898; cf. also G.P. 400,776, B., 1925, 7).—The fixing agents mentioned in the preceding patents, viz., sulphurised phenols, used either in the form of alkali salts as there described, or in the newly discovered form of colloidal solutions of the free phenols, are applied to the after-treatment of the coloured fabric in presence of dispersing agents such as soaps, proteins, sulphonic acids, etc. The addition of the dispersing agent causes a diminution of the affinity of the phenols for the fibre without affecting the lake formation between basic dye and phenols. The pattern on the fabric is therefore fixed fast to washing, whilst the ground is coloured only pale yellow. A. DAVIDSON.

Azo dyes (E.P. 256,808—9).—See IV.

Alkali salts of chloroimidodisulphonic acid (G.P. 431,116).—See VII.

Washing and bleaching agents (G.P. 429,855).—See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Materials of construction for nitrogen fixation. J. G. THOMPSON (Trans. Amer. Electrochem. Soc., 1926, **50**, 1—14. Advance copy).—The primary requirement of a steel to resist the action of nitrogen and hydrogen in the synthesis of ammonia at 100 atm. and 500° is a chromium content of at least 2%, with not more than one-seventh that amount of carbon. Nickel, tungsten, or vanadium may be added to improve the physical properties of the steel, but these cannot replace chromium (cf. B., 1925, 754). In the fixation of nitrogen as sodium cyanide at 1050°, cylinders of special nickel alloy withstand satisfactorily the oxidising action of the gas heating flames and also the corrosive action of the hot carbonate–cyanide charge. Catalyst bombs, in which a catalyst is maintained at 500°, are insulated by a Pyrex glass liner, which is inserted next to the bomb wall and keeps the temperature of the wall below 300°, thus preserving its full tensile strength. Chrome steels and chrome-plated steels appear to withstand the action of ammonium nitrate and ammonium carbonate in the synthesis of carbamide, and of gaseous mixtures of oxides of nitrogen and nitric acid at 200° in ammonia oxidation. The all-glass diaphragm gauge (B., 1923, 1 A) is useful for measuring pressures not exceeding 3—4 atm. where the gas attacks ordinary gauges. The magnetic automatic reducing valve of Larson and Karrer (B. 1923, 12 A) has proved useful. Illustrations of this valve, and also of high-pressure tubing and valves, are given. W. G. CAREY.

Electrolytic preparation of pure potassium hydroxide from crude potash. L. A. VERNITZ (Trans. R. Inst. Appl. Chem. [Russia], 1925, No. 4, 11—16; Chem. Abstr., 1926, **20**, 2621).—A wooden cell, coated with paraffin, is employed. During the production of potassium amalgam from potassium carbonate solution (18—30%, preferably the higher concentration) at the ordinary temperature, the anode consists of a platinum wire; the cathodic mercury must be continually renewed, the more rapidly the higher is the current density. The conversion of the potassium into hydroxide is effected electrolytically, using a nickel cathode; the concentration of the potassium hydroxide solution is allowed to increase up to 25—30%. The yield was 45—46%; with 15 amp. and 4·6—4·7 volts, 4·3 kw.-hrs. per kg. of potassium hydroxide were required. A. A. ELDRIDGE.

Producing bleaching powder by the Backman process. G. ANGEL (Chem. Met. Eng., 1926, **33**, 460—464).—The mechanical apparatus used (cf. G.P. 404,768; B., 1925, 207) is similar in principle to a pyrites furnace, and consists of a reinforced concrete tower with four intermediate floors and a central vertical shaft provided with specially treated iron scraper arms which cause the lime, fed from silos, to move from floor to floor in counter current to the chlorine, which is introduced at the second and third floors from the bottom to avoid the occurrence of mechanically bound chlorine in the finished product. Cooling coils are embedded in the intermediate floors, and cold water or refrigerated brine is circulated, the cooling being increased in the lower floors as the chlorination is completed. Regulation of the working is effected by changing the speed of the scraper arms, altering the clearance from the floor if necessary, also by temperature readings, and an analysis every 12 hours. The average content of chlorine in the bleach is 36—38%, with a maximum of 40%, and chlorine gas as low as 10% Cl can be utilised. The plant has been run for six months without interruption, and the life of the scraper arms is about six months in the lower floors and several years in the upper floors. W. G. CAREY.

Deterioration of strong sodium hypochlorite solutions. R. L. WELLS (Amer. J. Pharm., 1926, **98**, 404—406).—Examination over a period of three months of solutions of sodium hypochlorite containing 8·42—13·28% of available chlorine showed that the higher the concentration the higher is the rate of deterioration until a concentration of about 6% is reached. No substances were found which retarded the rate. Ethyl alcohol and aniline decomposed the sodium hypochlorite almost immediately and aluminium and copper also had a pronounced effect. E. H. SHARPLES.

Production of magnesia from dolomite. L. KIEPENHEUER (Zement, 1926, 15, 471—475; Chem. Zentr., 1926, II, 1088).—Calcined dolomite is compressed and mixed with water containing alkali carbonate and gases containing carbon dioxide are brought in contact therewith. The magnesium is obtained as a double carbonate in solution, which is decomposed, either *in vacuo* at 30° or at ordinary pressure at 95°, giving a precipitate of magnesium carbonate ($MgCO_3,3H_2O$), containing 1—4% of calcium, which can be utilised as the "magnesia alba" of commerce. The alkaline solution and surplus carbon dioxide are returned to the process. W. G. CAREY.

Production of alumina from alunite. J. YAMAZAKI and J. FURUKAWA (J. Soc. Chem. Ind. Japan, 1926, **29**, 147—153).—Alumina (99·65% Al_2O_3, 0·09% Fe_2O_3, 0·24% SiO_2) is prepared from alunite by treatment with sulphurous acid. One part of the alunite, which is previously ignited at 550° for 3 hrs. and powdered, is mixed with 10 pts. of water and treated with sulphur dioxide at 65°; more than 90% of the alunite is dissolved. The clear solution obtained contains aluminium sulphate and sulphite and potassium sulphate. When it is heated at 110°, sulphur dioxide is evolved and in the early stages $5Al_2O_3,3SO_2,15H_2O$ is precipitated, but after prolonged heating the whole of the aluminium in the solution is precipitated as $5Al_2O_3,3SO_2,2[K_2SO_4,Al_2(SO_4)_3],24H_2O$; the separation of aluminium and alkali in this way is therefore impossible. By adding an equivalent amount of ammonium or calcium sulphite to the solution and heating at 110°, over 94% of the alumina is precipitated as $5Al_2O_3,3SO_2,15H_2O$ in an easily filterable form; ammonium and potassium sulphate are recovered from the solution. After drying the basic aluminium sulphate thus obtained is heated above 900°; pure alumina is then produced accompanied by the evolution of sulphur dioxide. By dissolving the aluminium sulphite in sulphuric acid, pure aluminium sulphate is produced almost free from iron. K. KASHIMA.

Carbide furnace balance. J. BAUMANN (Chem.-Ztg., 1926. **50**, 629—631).—The theoretical amount of current required for the formation of carbide, as calculated from the materials, is 285 kw. per 100 g. of 85·5% carbide, but from experiments the author found that 395 kw. were needed, working with a 4000 kw. 3-phase furnace. A table is given showing the amounts of energy consumed (as calories and as percentages) due to carbon monoxide formation, production of slag by the action of calcium oxide and silicic acid, the endothermic heat of formation of carbide, the heat of fusion of carbide, the sensible heat loss from molten carbide run off and from waste gas and cooling water, and radiation losses. A quantity (9·92%) of metallic calcium was found in the carbide powder, which must be produced by dissociation of carbide in the furnace owing to overheating and volatilisation of the calcium. This secondary reaction was responsible for a consumption of energy of about 12%. When working with ingot furnaces this loss and also the sensible heat loss of the molten carbide can be almost eliminated, but this type of furnace has disadvantages in other respects. The author considers that, with the present type of furnace from which molten carbide is run off, there is little prospect of an improved energy balance. W. G. CAREY.

Action of steam and hydrogen on iron sulphide. F. FISCHER and H. TROPSCH (Abh. Kennt. Kohle, 1925, **7**, 178; Chem. Zentr., 1926, II, 1452).—On treating iron sulphide with hydrogen in the presence of steam, a small proportion of the sulphur combined with the hydrogen, the steam apparently exerting an oxidising action, but in the absence of steam no hydrogen sulphide was formed. L. A. COLES.

Determination of calcium sulphate in golden antimony sulphide. A. CHIAPPERO (Giorn. Chim. Ind. Appl., 1926, **8**, 1020).—Exactly 1 g. of the substance is stirred occasionally with 450 c.c. of water for 30 min., and the residue then collected on a tared Gooch or alundum crucible, washed until free from calcium, dried, and weighed. The loss represents calcium sulphate, on the assumption that other soluble substances are absent. T. H. POPE.

Manufacture of carbon disulphide. H. RABE (Chem.-Ztg., 1926, **50**, 609—611).—For the manufacture of carbon disulphide by the old method a cast-iron retort is used, since fireclay is permeable at the high temperature employed. The retorts are about 3 m. high, elliptical in shape, and have walls 4—8 cm. thick. If direct firing is used, the retort is fitted with a refractory jacket; with producer-gas firing this can be dispensed with if the retort is working normally. The charge is from 4—6 tons and a temperature of 1100° is reached, while according to Deish there is a loss of 18% of sulphur and 50% of charcoal. Explosions often take place owing to ignition of mixtures of carbon disulphide and oxygen; these and the weight of the charge cause the retort to bulge, so that the life of a retort is about 3 months, or at most 15 months. If the reaction temperature is not carefully controlled, secondary products may be formed, and sublimed sulphur may choke up the condenser tubes. For Taylor's electrical process cheap current is essential. The electric furnace used is over 16 m. high, the substructure, in which electrodes of resistance graphite are situated, occupying 4 m. of this height. The temperature of the resistance arc is considerably higher than that necessary for the reaction, and the formation of carbon disulphide takes place in the upper part of the furnace. Owing to the height of the furnace, its life is short and radiation losses are high, but the manufacturing costs are about equal to those of the old process. In the process of the Chem. Fabr. Griesheim-Elektron the charcoal is heated in a shaft between a fixed electrode and a movable one, the charcoal

serving as resistance. In the apparatus of Schultz the charcoal is raised to the necessary temperature by resistances separated from the charcoal; by this means fluctuations in current are avoided, the electrical connexions are not attacked by the gases, and the whole body of the charcoal is uniformly heated. The retort measures 2 by 3 m. and is 2¼ m. high for an output of 500—700 kg. of carbon disulphide, while the consumption of power is 30 kw. for 1000 kg. of carbon disulphide, the efficiency being 75—80%. This furnace is more under control than the older types, working costs are lower, and the heating efficiency is higher.

W. G. Carey.

Extraction of bromine from salt liquors. A. Moreschi (Giorn. Chim. Ind. Appl., 1926, **8**, 115—116).—Brine concentrated to d 1·21 is treated with chlorine, and the bromine thus liberated extracted continuously by means of a solvent, carbon tetrachloride being especially suitable. More than 60% of the total bromine is recovered if this extraction is effected in an emulsor. The carbon tetrachloride solution of bromine may be separated from the salt liquor and from its emulsions with these liquors by forcing the emulsions through a capillary system. The bromine may then be recovered almost quantitatively from the carbon tetrachloride solution by treatment with lime suitably hydrated, the reaction being rapid and capable of being rendered continuous. The product thus obtained is a pulverulent substance, which yields all its bromine when treated with a dilute acid and is termed " bromide of lime."

T. H. Pope.

Measurement of oxygen at high pressures. Q. Sestini (Giorn. Chim. Ind. Appl., 1926, **8**, 117—119). —The errors introduced by assuming that oxygen obeys Boyle's law at the high pressures prevailing in cylinders of the compressed gas are calculated. [The numerical data given agree moderately closely with those published in Appendix VII of the First Report of the Gas Cylinders Research Committee, 1921; cf. J.S.C.I., 1922, 37 r.]

T. H. Pope.

Patents.

Stabilisation of liquid hydrocyanic acid. M. Walker, Assr. to Pacific R. & H. Chemical Corp. (U.S.P. 1,591,899 and 1,591,900, 6.7.26. Appl., 16.4.24 and 4.8.25).—(a) Liquid hydrocyanic acid may be kept for several months at a comparatively high temperature and pressure, without decomposition or polymerisation, by slightly acidifying it with sulphuric acid and introducing into the container a metal which readily forms a complex with ammonia, *e.g.*, copper, cobalt, or monel metal, in the form of strips, gauze, or powder. Copper is most effective, as it is dissolved to an appreciable extent by liquid hydrocyanic acid. (b) Nickel is used in place of the other metals mentioned.

R. B. Clarke.

Apparatus for analysing [oleum] by heat of reaction. J. C. Boertlein, Assr. to Grasselli Chemical Co. (U.S.P. 1,594,593, 3.8.26. Appl., 1.3.24). —Sulphuric acid of known concentration and oleum are fed at a constant rate into the top of two cylindrical vessels, provided with sediment traps, connected at the bottom by a Y-tube which projects into the mouth of a Dewar flask. Thermometers are placed inside the latter and inside each cylinder.

R. B. Clarke.

Producing sulphur dioxide and apparatus therefor. H. O. C. Isenberg, Assr. to Gen. Chemical Co. (U.S.P. 1,595,196, 10.8.26. Appl., 22.12.24).—The apparatus contains a supply of molten sulphur, the upper layers of which are fed to the burner, whilst the lower layers, containing impurities which have settled out by gravity, are withdrawn periodically. Portions of molten sulphur are also withdrawn at intervals from the lower layers of the sulphur in the burner.

L. A. Coles.

Testing liquid sulphur dioxide. [Determination of moisture content]. W. C. Dever, Assr. to Kelvinator Corp. (U.S.P. 1,597,513—5, 24.8.26. Appl., 1.8.25).—(a) The apparatus is a glass container with a liquid level line and a lower prolongation of reduced section graduated from about one ten-thousandth to above one-thousandth of the volume to the level line. (b) The method consists in evaporating a measured volume of the liquid at normal temperature without access of air, measuring the volume of the residual water, absorbing and weighing the water vapour carried off by the gas, and calculating the percentage of total water by reference to the specific gravity of liquid sulphur dioxide. (c) The graduation of the apparatus (a) is such as to compensate for the water vapour carried off and to indicate directly the required percentage by weight of total water.

H. Holmes.

Extraction of salts from aqueous solutions. A. J. Stephens. From Salt Production Synd., Ltd. (E.P. 256,294, 1.4.25).—Sea water is concentrated at a high temperature until nearly saturated with calcium sulphate, *i.e.*, to about one-third of its volume, evaporation is continued in another evaporator maintained at about 38° until the calcium sulphate is completely precipitated, and evaporation is continued in a third vessel to crystallise out the salt. Each stage of the evaporation is conducted under reduced pressure in pans heated by calandria. In the second stage, evaporation is effected in one or more shallow pans arranged in tiers, heated by calandria of limited depth. The liquid from the first evaporator is delivered directly into the second evaporator, but the liquor from the first and second stages flows out of the bottom of the vessels into sumps in which calcium sulphate and sodium chloride settle out respectively, the clear liquor from which the calcium sulphate has settled out being drawn into the third evaporator by suction. Each calandria is heated by the vapour evolved in the same stage of the process, the vapour being raised to the required temperature by compression; live steam is admitted simultaneously into the first calandria, to compensate for heat losses during the process.

L. A. Coles.

Density regulator for concentrating evaporators [for brine]. J. Price, Assr. to Griscom-Russell Co. (U.S.P. 1,595,244, 10.8.26. Appl., 28.11.21).—Two liquid-containing towers communicate directly with the lower part of the evaporator shell, and means are provided for admitting small quantities of fresh water to the upper end of one column. Floats in the brine column

and the fresh water column control the admission and the discharge respectively of the liquid in the evaporator.
H. Holmes.

Process of manufacturing arsenates. J. D. Jenkins and E. F. Berger, Assrs. to Pittsburgh Plate Glass Co. (U.S.P. 1,596,662, 17.8.26. Appl., 21.8.24).—White arsenic is dissolved in a weak alkali solution, and the alkali arsenite thus formed is oxidised to arsenate in the presence of copper in the solution as a catalyst.
H. Royal-Dawson.

Manufacture of tin silicofluoride. Soc. de Récherches et de Perfectionnements Ind. and Soc. d'Electro-Métallurgie de Dives (F.P. 606,541, 20.2.25).—Tin is treated with hydrofluosilicic acid in the cold or at gentle heat, in presence of a limited amount of a catalyst such as nitric acid, ammonium nitrate, or other oxidising agent. W. G. Carey.

Regeneration of the electrolytic liquor from the electrolytic production of perborate. Henkel & Co., Assees. of M. Jacobi (G.P. 431,075, 6.9.25).—At suitable intervals the solution is boiled with silica gel, and then again separated from the gel. W. G. Carey.

Production of alkali salts of chloroimidodisulphonic acid. F. Raschig (G.P. 431,116, 22.4.25).—Alkali salts of imidodisulphonic acid are treated with hypochlorous acid. Thus potassium imidodisulphonate is dissolved by gently heating in a solution of sodium hypochlorite, and the solution decomposed with dilute acetic acid. Alternatively solutions of sodium hypochlorite and sodium imidosulphonate are mixed and the mixture is treated with glacial acetic acid and diluted. Acetic acid may be replaced by other acids which are not oxidised. The reaction occurring is $NH(SO_3M)_2 + M'OCl = NCl(SO_3M)_2 + M'OH$. In contrast to the potassium salt, the sodium salt of chloroimidodisulphonic acid is very readily soluble in water. The products find technical application as disinfecting and bleaching agents. J. S. G. Thomas.

Production of chrome alum from solutions of ferrochromium. H. C. Starck Kommanditges. auf Aktien, F. Klaus, and R. Basler (G.P. 431,201, 12.8.23).—The ferrochromium is dissolved in sulphuric acid, and the solution, before or after precipitation of part of the ferrous sulphate with potassium sulphate, treated with potassium dichromate in the cold.
J. S. G. Thomas.

Preserving the pulverised condition of calcined soda during storage. J. Urchs (G.P. 431,256, 10.8.24).—Sodium carbonate is intimately mixed with a small proportion of borax meal. J. S. G. Thomas.

Recovery of lithium carbonate from residual liquors containing potassium sulphate. Metallbank u. Metallurgische Ges. A.-G., Assees. of H. Weidmann (G.P. 431,257, 30.5.25).—Potassium sulphate is salted out by addition of potassium salts, more especially potassium chloride, and lithium is then precipitated as lithium carbonate. The mother liquor containing potassium chloride is used to precipitate potassium sulphate from a new charge. J. S. G. Thomas.

Apparatus for containing hot solutions containing both ammonium chloride and ammonium nitrate. I. G. Farbenind. A.-G., Assess. of H. Rassow (G.P. 431,508, 6.4.23).—The apparatus is constructed of chromium-nickel steel with a low carbon content. An alloy containing Co 20%, Ni 6% is attacked less readily by boiling saturated ammonium chloride solution containing 20% of ammonium nitrate than by a solution containing ammonium chloride alone. L. A. Coles.

Production of ammonium chloride crystals. I. G. Farbenind. A.-G., Assees. of W. Eissner (G.P. 431,760, 18.12.23).—Biuret or cyanuric acid, or a mixture of one of these with the other or with glycerin, is added to ammonium chloride solutions containing excess of acid or ammonia, whereupon ammonium chloride separates in cubic crystals. L. A. Coles.

Burning limestone. J. K. Kiddle (E.P. 256,687, 12.5.25).—Limestone ground to 40-mesh size is heated to a temperature between 700° and 900°, preferably in a rotary electric muffle furnace in the presence of an oxidising agent or superheated steam.
H. Royal-Dawson.

Manufacture of titanium oxide. P. A. Mackay (E.P. 256,734, 28.5.25).—Oleum is added gradually to damp pulverised ilmenite, at such speed that the temperature of the reaction rises slowly while the mass is stirred continuously; the product is treated in known manner to separate the desired titanium salt or oxide.
H. Royal-Dawson.

Removal of hydrogen sulphide from gases. I. G. Farbenind. A.-G., Assees. of S. Schneider and F. Schweitzer-Hennig (G.P. 431,307, 3.9.24).—Gases containing hydrogen sulphide are brought into intimate contact at 70—75° with a solution containing sodium sulphite and sodium hydrogen sulphite in molecular proportions, whereby a solution of pure sodium thiosulphate is produced. J. S. G. Thomas.

Furnace (U.S.P. 1,590,161).—See I.

Utilisation of material containing lime and bitumen (G.P. 431,255).—See II.

Cement and by-products (U.S.P. 1,594,689).—See IX.

Removing iron from materials (E.P. 256,428).—See X.

VIII.—GLASS; CERAMICS.

Fining of pure and aluminous alkali-lime-silicate glasses with saltcake or arsenic. E. Zschimmer, E. Zimpelmann, and L. Riedel (Sprechsaal, 1926, **59**, 331—333, 353—357, 393—395, 411—413, 422—425).—The effect on the melting of the glass SiO_2 74%, CaO 13%, Na_2O 13%, and on glasses derived therefrom by the replacement of silica by 1 to 12% of alumina, of additions of saltcake (with or without nitre), and arsenic with nitre were observed. The simple soda-lime glass was fined best by addition of 0·2% Na_2O as sulphate. With increase of alumina tendency to foaming increased. With 0·2% of soda as sulphate the parent glass was best fined according to the time-temperature scheme (after the last filling on) :—1350°—1480° (1 hour)—1280° (2 hours), when 100 g. of glass were melted. During the period of rise of temperature the saltcake reacted with silica, and the SO_3 so formed

was largely dissociated, the resulting gases performing the fining operation. The subsequent rapid drop of temperature prevented further formation of SO_3. Arsenious oxide and nitre acted in the same manner as saltcake, producing frothing when used in excessive amounts. Saltcake could not be used for fining highly aluminous glasses (over 6% Al_2O_3), but these were successfully freed from seed by the use of arsenious oxide and nitre. Samples of the glasses formed were photographed with " ultramicroscopic " illumination.

A. COUSEN.

Temperature–time curves of the visible devitrification of plate glass. E. ZSCHIMMER and A. DIETZEL (Z. tech. Physik, 1926, **7**, 278—282; Chem. Zentr., 1926, II, 1320).—A devitrification apparatus with a crucible thermo-element is described which permits test-pieces of glass of 0·05 g. weight to be heated rapidly to the devitrification temperature and cooled with simultaneous exact temperature and time measurements. Phenomena so observed in the devitrification of Waldhof plate glass are described. Maximum lengths of the crystals were measured in thin sections of the devitrified samples treated over various temperatures and times. The rate of crystallisation was fixed, and the curve showing its relationship to the temperature plotted; the maximum lay at 1005°, at which temperature crystals 20 μ in length formed in one minute. Temperature and time curves for crystal lengths of 10, 50, and 100 μ are discussed. In the above-named samples the crystals obtained at 1000° were identified by *X*-ray methods as tridymite and wollastonite.

A. COUSEN.

Signal green and the absorption of copper oxide in glasses of various compositions. E. ZSCHIMMER, C. M. GRISAR, and H. MEESS (Z. tech. Physik, 1926, **7**, 290—300; Chem. Zentr., 1926, II, 1320).—A systematic series of glasses containing the oxides of silicon, boron, aluminium, sodium, potassium, calcium, magnesium, zinc, and lead was prepared, and the influence of 4·5 % of copper oxide on the transmission of light of various wave-lengths determined. All curves of the transmission factors for samples 1 mm. thick gave a maximum transmission in the region of wave-length 500 $\mu\mu$. The position of the maximum may be considered as a special property of the copper oxide, which apparently occurred in the glass as silicate or borate, with, in some cases, a partial reduction to give a ruby colour. All the glasses absorbed strongly in the red region 700 $\mu\mu$, and the proportion of red transmission to that of the blue-green (500$\mu\mu$) was dependent to a great degree on the nature and proportions of the glass constituents. For each melt the plate thickness for a 2% red transmission was calculated as well as the transmission factors for green, 530 $\mu\mu$, and blue, 450 $\mu\mu$.

A. COUSEN.

Detection of cadmium and arsenic in glass. W. GEILMANN (Glastech. Ber., 1926, **4**, 92—97).—The method, based on that of Biewand, consists in placing a mixture of powdered glass and anhydrous sodium oxalate at the closed end of a glass tube, which is then drawn into a capillary above the powder. By suitably heating the mixture a metallic sublimate is deposited in the cold capillary. It is possible to detect 0·0025% of cadmium in 0·1 g. of glass or by microchemical means 0·1% in 2 mg. The same procedure may be used for testing for arsenic in glass, whereby 0·0025% may be detected in 0·1 g.

A. COUSEN.

Constrained crystallisation of glasses. J. F. PONOMAREV (Z. anorg. Chem., 1926, **155**, 281—290).—The true m.p. of a variety of glasses containing sodium and boron have been determined by the method previously employed (A., 1915, ii, 449). Borax–alumina and borax–calcium phosphate mixtures are recommended for use as readily fusible lead-free glazes. R. CUTHILL.

New glass stone: $Na_2O,3CaO,6SiO_2$. A. B. PECK (J. Amer. Ceram. Soc., 1926, **9**, 351—353).—Stones formed by devitrification in glass sheets drawn from the tank were identified, on the basis of Morey and Bowen's work on the system Na_2SiO_3–$CaSiO_3$–SiO_2 (B., 1925, 921), as the compound $Na_2O,3CaO,6SiO_2$. The optical properties of the crystals are given. The decomposition point of the compound is 1047°, and since most of the compounds in this system are somewhat unstable, a slightly higher temperature in the drawing chamber would probably eliminate them from the glass.

F. SALT.

Quartz sand and massive quartz, two different mineralogical modifications. T. Schauer (Sprechsaal, 1926, **59**, 473—475; Chem. Zentr., 1926, II, 1321).—German crystal sand is more difficult to invert to cristobalite than Norwegian quartz. The solubilities of cristobalite, massive quartz, and crystal sand in hydrofluoric acid under fixed conditions are as 2·6 : 55·6 : 70·6. The variation in transparency of these substances in porcelain results only from the greater solubility of the inverted cristobalite in the molten felspar. When sand and massive quartz are melted and then inverted at 1470° into cristobalite, they act identically, and the rapid cooling of the glass so formed is favourable for inversion. The author considers that quartz obtained from the melt by slow cooling is β-quartz, which is the cause of the difficult solubility, and which occurs in the case of German sands. He holds that the quartz obtained by rapid cooling is α-quartz, as occurs in massive quartz. This is more easily inverted than β-quartz.

A. COUSEN.

Bacteria in earthenware bodies. A. SCHOBLIK (Keram. Rundsch., 1925, **33**, 893).—An earthenware body was examined microscopically after having been " aged " for varying periods. Bacteria were observed in large numbers after a short time; a white scum appeared on the filter-cakes after about two weeks. Colonies of algæ were also observed in places, but they were scarcely in sufficient quantity to affect plasticity. Rod bacilli were found to be present in the clay in large numbers during the first part of the weathering period. These were later displaced almost completely by *Diplococci* and *Tetracocci*. The latter were obtained as a pure culture by repeated inoculation on a nutrient medium of peptone agar and finely ground highly plastic clay. The *Diplococci* adhered in large numbers to the surfaces of the clay particles, and when cultivated in an earthenware body they were found to increase the

plasticity. This property is apparently affected by bacterial metabolism. F. SALT.

Drying of ceramic ware. W. PUKALL (Sprechsaal, 1926, **59**, 367—370; Chem. Zentr., 1926, II., 1317).—Moisture can be raised from a depth of 9·5 in. by simple evaporation at the porous surface of a Pukall filter. In fine-pored materials only capillarity comes into play on drying, and this goes on only at the outer surface. In large-grained masses the drying may also occur from the surface towards the interior. The rate of drying of fine-pored and fine-grained materials is consequent upon external atmospheric pressure, and such materials are dry within while still moist at the surface. The drying process is independent of atmospheric pressure. Cooling and a reduced internal pressure results from the evaporation of the water. Drying is dependent upon the temperature and humidity of the air. The b.p. of the internal water, under negative pressure, need not be exceeded by the application of artificial heat. The most suitable conditon is that in which the vapour pressure within the material approaches that holding externally. Drying goes only so far that internal and external air pressures are equal. Dryness can be attained by means of porous materials capable of absorbing moisture as well as by the air. A. COUSEN.

Electrical resistivity of some ceramic materials at elevated temperatures and a simple commercial method for its determination. R. M. KING (J. Amer. Ceram. Soc., 1926, **9**, 343—350).—Sufficiently accurate readings were obtained by the use of a high-resistance voltmeter, satisfactory contact between the refractory test-pieces and the metal terminals being secured by carefully grinding the surfaces of the samples. The use of molten metal terminals, or of a layer of lampblack, was precluded because, on the one hand, the test-pieces were porous, and on the other, oxidising conditions were desired. Results are tabulated for a number of materials, including chemical porcelain, talc, sillimanite, etc. Periclase and Chinese talc gave the highest resistances, followed by magnesia spinel, cyanite, sillimanite, and andalusite. The data indicate that, by substituting MgO for Al_2O_3 or SiO_2 in the composition Al_2O_3,SiO_2, the electrical resistance will increase as the magnesia content rises. F. SALT.

Green scumming [on fireclay]. E. R. CURRY (J. Amer. Ceram. Soc., 1926, **9**, 392—397).—A greenish-yellow efflorescence, which appeared on the surface of fireclay ware, was found on analysis to contain 7·7% V_2O_5 and 1·0% of nickel oxide. The addition of lead and bismuth salts to the body mixture did not entirely prevent the formation of scum, whilst antimony and zinc compounds had no effect. Scumming was effectively prevented, however, by dipping individual pieces of ware in a solution of 30 g. of barium chloride and 30 g. of ammonium chloride in one gallon of water. F. SALT.

Variation of the "cone" squatting point of whiteware glazes with change of chemical composition. E. ZSCHIMMER and E. LEONHARDT (Z. tech. Phys., 1926, **7**, 287—290; Chem. Zentr., 1926, II, 1317—1318).—A cone-squatting point "*vn*" characteristic of the softening of glasses was defined; thus the point $t°vn$ for a particular glass was the temperature at which a pyramid of the shape of an industrial Seger cone, heated at a rate $v = n°$ per min., squatted so that the tip came into contact with the base plate. The cone squatting points for two whiteware frits with differing compositions and rates of heating were determined. Lime used in glaze frits raised the squatting point considerably. By determining the squatting point of glasses of varying composition for various rates of heating, the change of viscosity with temperature will be indicated. A. COUSEN.

Interferometer measurements of the thermal dilatation of glazed ware. G. E. MERRITT and C. G. PETERS (J. Amer. Ceram. Soc., 1926, **9**, 327—342).—The unreliability of measurements taken on glaze specimens cast or drawn from the molten glaze batch alone is demonstrated. An interferometric method developed at the Bureau of Standards (Sci. Papers 393 and 485) for measuring the thermal expansion of specimens ranging from 0·2 to 10·0 mm. in length between 20° and 1000° was used to obtain a comparative series of measurements on samples of body and glaze taken from the finished ware ("glaze" specimens) and on cast and drawn samples of the glaze alone. Six different glaze mixtures were tried. The "glaze" specimens were fragments of glaze broken from the fired ware and freed from adhering pieces of body. All the specimens were subjected to two identical heating and cooling treatments. The curves for the first heatings furnished valuable information regarding the previous contraction of the ware during manufacture, whilst the curves for the second heatings gave the expansions of the materials after they had all received approximately the same heat treatment as the result of the first runs. Comparison of the two sets of curves (first and second heatings) shows that the thermal expansion of a given sample is affected by the heat treatment it has previously received. The curves for the second heatings show marked discrepancies between the "glaze" specimens and the cast and drawn samples. The last two are, therefore, not reliable guides. This is further demonstrated by taking expansion measurements of both body and glaze taken from a badly crazed plate, and comparing these data with measurements of cast and drawn samples of the same glaze batch. On heating glazes or glasses a critical region of about 75° occurs between 450° and 600°, depending upon the composition. Stresses are liable to accumulate in glazes at about 20° above the beginning of the critical region, or near the upper limit of the annealing range. A differential contraction of about 5 microns per cm. between body and glaze is sufficient to cause crazing. F. SALT.

PATENTS.

Colourless Crookes' glass. CHANCE BROS., & CO., LTD., P. V. W. GELL, C. E. GOULD, W. M. HAMPTON, and H. S. MARTIN (E.P. 256,737, 5.6.25).—A glass, efficient in absorbing ultra-violet rays and practically colourless, is obtained by the addition of two or more ingredients, all of which absorb ultra-violet rays, and the colours produced by them neutralise one another. As an example a glass containing approximately 3·3% of cerium and 0·7% of didymium (both preferably added as oxides

or hydroxides) is practically colourless and an efficient absorber of ultra-violet rays. A. COUSEN.

Making glass. C. E. PARSONS, Assr. to METALS RESEARCH CORP. (U.S.P. 1,595,358, 10.8.26. Appl., 19.9.25).—Glass is made in a blast furnace by using a charge consisting of a mixture of an alkali chloride with carbonaceous material and a blast-furnace slag. The charge is ignited and melting continued until a glass free from iron and consisting essentially of calcium sodium silicate is produced. B. W. CLARKE.

Glass melting furnace. L. MAMBOURG, Assr. to LIBBEY-OWENS SHEET GLASS CO. (U.S.P. 1,596,058, 17.8.26. Appl., 7.2.23).—Molten glass is introduced into a fining tank which is subsequently united to a melting tank charged with batch, the glasses in the two chambers then being caused to unite into a single pool. Provision is made in the case of a continuous tank furnace connected to a refining tank by a neck or conduit, for moving away and breaking the connexion of the melting furnace with the fining chamber. A. COUSEN.

Continuous compartment kiln. A. V. BLEININGER, Assr. to HOMER LAUGHLIN CHINA CO. (U.S.P. 1,595,817, 10.8.26. Appl., 10.9.23).—The kiln consists of a number of contiguous compartments each provided with a removable platform for supporting the ware to be fired, and a combustion chamber communicating with the interior of the compartment. Each combustion chamber also communicates with the interior of an adjacent compartment so that the hot products of combustion may be passed from one compartment to another, and air flowing through one compartment for cooling purposes can be used for supporting combustion in the adjacent compartment. B. W. CLARKE.

Leadless enamels. C. TOTOT-GIBARU (Addn. 30,444, 1.5.25, to F.P. 597,146; B., 1926, 323).—The metal is first coated with a powdered mixture containing, *e.g.*, 22·63% SiO_2, 3·76% Al_2O_3, 3·5% CaO, 23·44% ZnO, 16·6% Na_2O, 2·63% K_2O, 12·5% Sb_2O_3, 13·72% B_2O_3, and 2·02% F, then with a similar mixture containing, *e.g.*, 15·06% SiO_2, 1·08% Al_2O_3, 12·65% CaO, 18·48 Na_2O, 30·12% ZnO, 21·15% B_2O_3, and 2·43% F. A. R. POWELL.

IX.—BUILDING MATERIALS.

Reducing conditions and colour changes in the burning of Portland cement. H. KÜHL [with W. ADAM] (Zement, 1926, **15**, 456—458; Chem. Zentr., 1926, II., 1322).—The iron compounds of Portland cement may be converted by reducing agents into the metal. The yellow, brown, and red colours of the clinker are not removed on reduction. In sintered clinker the ferric oxide is free since ferrites decompose above 1400°. The presence of ferric oxide is conditioned by rapid cooling, ferrites resulting on slower cooling, and when these are not formed the ferric oxide colours the material. A. COUSEN.

Grain size of Portland cement and its influence on the rate of hydration. A. HAUENSCHILD (Zement, 1926, **15**, 453—456, 469—471, 488—492; Chem. Zentr., 1926, II., 1322).—The sulphates of the clinker and also gypsum, added to regulate the setting time, are concentrated in the finest fractions. The grain size of the cement is distinguished otherwise only by loss on ignition, which is greatest in the finest fractions and decreases with increasing grain size. Setting time decreases with increasing fineness. The weight per litre considerably decreases with increasing fineness. Constancy of volume is increased by fine grinding. The greatest strengths are reached with grain sizes of 19·7 μ to 36·3 μ. Fine powder with grain size below 7 μ is decomposed by atmospheric moisture and carbon dioxide soon after grinding and therefore does not give the greatest strength. Hydration occurs through the action of moisture on the surface of the grains and its velocity increases with decreasing grain size. Grains less than 15—18 μ are completely hydrated, all clinker-forming minerals being practically completely changed. Larger grains are only partly hydrated. Alite hydrates appreciably more rapidly than the other clinker-forming minerals. Hydration, further, is dependent on the size of the clinker crystals, and a simple crystal of 15 μ diameter is changed more rapidly than a grain of equal size composed of various clinker-forming minerals. A. COUSEN.

Blast-furnace cement. H. BURCHARTZ (Zentr. Bauverwalt., 1926, 241; Mitt. Materialprüf., 1926, 62—63).—The results of ten-year tests on the tensile strength, crushing strength, and variation in length of mortar and concrete made with blast-furnace cement fully bear out the results obtained for the shorter periods, and compare very favourably with those given by Portland cement and iron Portland cement. B. W. CLARKE.

Deterioration of [timber] structures in sea water. Experimental work of F. D. White and C. M. Yonge at Plymouth during July and August, 1924. G. BARGER (Sixth Interim Report, Comm. Inst. Civ. Eng., Dept. Sci. Ind. Res. 1926, 9—13; cf. B., 1925, 761).—Compounds of the phenarsazine (D.M.) type are the most toxic towards *Teredo* larvæ, the toxicity appearing to depend on the arsenic with oxygen or halogen attached, together with two benzene nuclei. Fluorenone is very toxic towards *Limnoria*, but has relatively little effect on *Teredo*; organic arsenic compounds have little effect on *Limnoria*. A cheap specific against *Limnoria* has still to be found. Rafts have been moored at Plymouth constructed of timber impregnated with creosote and with phenarsazine, and other toxic compounds to test the protection afforded against *Teredo* and *Limnoria*. B. W. CLARKE.

PATENTS.

Production of plastic masses for use as paving materials. E. C. R. MARKS. From BENZOL-VERBAND GES. M.B.H. (E.P. 256,513, 15.3.26).—Tar, pitch, oils, coumarone resin, or similar materials are mixed with spent oxide from iron oxide purifiers in gas works and heated to bring about a vulcanising action, producing a non-adhesive hard and elastic mass which can be used as a base for artificial asphalt or tar macadam. B. W. CLARKE.

Method for calcining and clinkering [cement] with recovery of by-product heat and by-products. R. D. PIKE (U.S.P. 1,594,689, 3.8.26. Appl., 1.7.24).—The raw mix for making Portland cement clinker, after being calcined in a neutral atmosphere to minimise

oxidation of sulphur, is passed to an inclined rotary kiln in which the sulphur in the mix is oxidised to sulphur dioxide and sulphur trioxide in an oxidising atmosphere. If necessary, sulphur dioxide is introduced into the kiln by means of sulphur burners. The kiln gases containing oxides of sodium, potassium, and sulphur pass into a waste-heat boiler, through a humidifying chamber, and thence through an electrical precipitator from which the sulphates of sodium and potassium are collected. The clinker is removed from the kiln by means of a chute. R. B. Clarke.

Manufacture of fused cement. L. G. Patrouilleau, and Soc. Anon. Alumine et Dérivés (F.P.604,916, 21.10.25).—A mixture of raw materials is sintered in a refractory-lined rotary tube furnace, and the sintered mass fused on leaving the furnace. For this purpose the metal wall of the furnace in the region of the outlet is enlarged in the form of a disc and connected with one pole of a source of current of low tension and high current strength. The disc is set obliquely and can be adjusted by means of a hinge and spring with respect to a metal or graphite plate which is connected with the other pole of the source of current. The sintered mass falls between the disc and the plate, closing the current circuit and being melted by the current flowing. J. S. G. Thomas.

Preservation of wood. Höntsch & Co. (G.P. 430,642, 7.10.25).—Sweating of wood impregnated with acetone oil or with solutions containing it, is prevented by adding to the oil a quantity of barium, potassium, or sodium naphthenate insufficient to cause emulsification. L. A. Coles.

Burning limestone (E.P. 256,687).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Specific efficiency of the blast furnace. R. Franchot (Min. and Met., 1926, 7, 368—372).—In typical practice blast-furnace smelting efficiency is 42% with a 42% loss in gas and 16% loss in radiation and top heat, including moisture evaporation and other minor factors. Improvement of efficiency in iron making is to be sought in the utilisation of some of the latent energy of the carbon monoxide passing unused out of the furnace. The energy so lost is more than would be absorbed in reducing, melting, and slagging 100% increase of ore. Frequently not much more than 80% of the coke charged burns with air at the hearth, about 5% being absorbed by the iron and 15% is lost by "solution," that is, gasified otherwise than by air. If this last loss could be prevented, energy for 26% more smelting would be set free. Solution loss, which is a result of the surplus heat developed by the air, may be prevented by increasing the burden of the furnace and by withdrawing gas at a level a little above the hearth. The removal of the hot gas, which would carry with it cyanide vapour, would result in the saving of hearth heat consumed in the vaporisation and cyanidation of alkali accumulations and effect an adjustment between hearth and shaft, thereby substantially increasing iron production with the expenditure of less coke. The comparative efficiencies of several furnaces, derived from operating data, are considered, and it is shown that from the thermodynamic point of view benefit in all cases would accrue by withholding from the shaft a substantial proportion of the gas formed in the hearth. To balance the surplus energy in American furnaces with work done it would be necessary to withdraw about 44% of the gas formed from the coke burning with air. This would permit at least 12½% of the coke carbon gasified by ore and stone in the shaft to reach the hearth and burn with air. The sensible heat of the hot gas withdrawn would provide blast heat, and further, this withdrawal, by limiting the accumulation of saline matter in the furnace and by removing slag vapour, would lessen condensation in the shaft and the tendency to bridging, and so facilitate stock movement. The recovery of cyanides from the withdrawn gases and the potential utilisation of the blast furnace as a means of fixing atmospheric nitrogen are considered. M. Cook.

Rate of carbon elimination in the Martin [open-hearth steel] furnace. E. De Loisy (Rev. Mét., 1926, 23, 369—380).—In the normal Martin process where the bath is covered by a sufficiently large quantity of slag and with an excess of iron oxide, the speed of decarbonisation at any moment is proportional to the quantity of carbon remaining in the bath. The law established is expressed by $\log C/C_0 = -kt$, which does not give the rate of the ferrous oxide–carbon reaction, but the rate of diffusion of carbon in the metal until it comes in contact with the slag. The duration of the operation is proportional to the square of the depth of the bath. The results can be applied, other things being equal, to all reactions where a bath of metal is refined by a supernatant liquid or gaseous phase. M. Cook.

Martensite. H. Hanemann and A. Schrader (Trans. Amer. Soc. Steel Treat., 1926, 9, 169—239).—Needles of martensite contain less carbon than the austenite with which they are in contact. The heat of formation of martensite is not equal to the γ-α-iron heat effect. The interatomic distance in martensite is greater than in α-iron. These facts and the authors' quenching, tempering, and liquid air immersion tests on high-carbon steels lead them to infer that martensite is not α-iron, but consists of two previously unidentified phases of the iron–carbon system, ϵ and η, which are in metastable equilibrium with austenite. A constitutional diagram of the metastable system is drawn up, the equilibrium lines lying at a lower temperature than in the cementite system. By means of the electrocardiograph a critical point in pure iron was found at 685°, at which ϵ is formed from γ-iron. ϵ-Iron dissolves 0·10% C, whilst α-iron dissolves 0·006% C and also differs in tensile properties. 71 photomicrographs are given, from which the structure of martensite is interpreted in terms of the new hypothesis. The crystallisation of the ϵ-phase and ϵ-η-γ mixtures is the cause of the acicular martensitic formation. ϵ-Iron changes to α-iron on tempering with precipitation of Fe_3C. From 0·1—0·9% C the ϵ-phase decreases and the η-phase increases. Above 1·4% C η crystallises as needles in the γ-grains. The lower density of martensite is a property of the η phase, which commences to decompose at 80°. The η phase is a

compound containing the iron carbide $Fe_{24}C$, and the bearer of the martensitic hardness. Its maximum crystallising power occurs slightly below room temperature. Changes in the conductivity of quenched steels due to tempering result from decomposition of the η- and γ-phases. The relative position of the cementite and martensite systems is shifted in alloy steels.
T. H. Burnham.

Hardness of different structures in steel. K. Tamaru (Sci. Papers Inst. Phys. Chem. Res., 1926, **5**, 25—44).—The Brinell hardness at ordinary temperature of austenite and martensite in carbon steel is 155 and 720 respectively. At 820° the hardness of austenite in carbon steel is 10 on the Shore scale. The hardness of normal cementite is 640 Brinell, and in the strained condition 820. In manganese steel the hardness of austenite at both ordinary and high temperature is 182 and 17 on the Brinell and Shore scales respectively. In steels containing only a trace of carbon, austenite is obtained when the manganese content exceeds 16%, but in the presence of 2% of carbon, 5% of manganese suffices to produce an austenitic structure. The hardness of martensite increases as the length of the martensite needles diminishes. The variations in hardness of carbon-, nickel-, and manganese-steels with temperature have been measured. The hardness of tempered carbon steels at 112° is greater than that in the quenched state. As the tempering temperature rises further the hardness gradually diminishes, with the exception of three abnormal changes which take place at 150—200°, 350—400°, and 430—450° respectively. In water-quenched carbon steels the hardness increases with the carbon content up to 0·8% and afterwards gradually decreases.
M. Cook.

Carburisation by solid cements. W. E. Day (Trans. Amer. Soc. Steel Treat., 1926, **9**, 240—258).—The causes of irregularities in depth of case produced by solid cements even under strict control include the presence of hydrogen in the gases evolved by the carburising mixtures, which tends to exclude the penetration of carbon monoxide and has a strong decarburising effect under certain conditions. In tests on nickel- and nickel–chromium case-hardening steels, the concentration of carbon was made to exceed the saturation point by temperature oscillations such as may occur in careless control. By heating nickel steel *in vacuo* the diffusivity constant was determined, and was found to depend chiefly on the temperature. The theoretical curve for carbon penetration at 870° was found to agree with the actual curve within the limits of error. Diffusion may be used to produce a eutectoid case by slow cooling. Regenerative quenching sometimes suffices for this. The carbon concentration varies with the shape of the surface to be cemented.
T. H. Burnham.

Dimensional changes accompanying the phenomena of tempering and ageing of tool steels. H. Scott (Trans. Amer. Soc. Steel Treat., 1926, **9**, 277—304).—Experiments were made on cylinders of 1 in. diameter and 4 in. long of twenty carbon and alloy tool steels. The increase in size on quenching was mostly in diameter with surface-hardened steels, but mostly in length for fully hardened steel when the quenching temperature was low. On tempering, the changes in fully hardened steel were the same in length as diameter, but greater in length than diameter for surface-hardened cylinders. On tempering, the initial contraction was greater with water-quenching than with oil-quenching and with a high quenching temperature than with a low one, but the final contraction was not affected. The initial contraction was eliminated and the expansion intensified by interrupting the oil-quenching and cooling in air through the hardening transformation. All the quenched steels contracted on keeping. The contraction was the same for 1 hr. at 100° as for 6 months at room temperature. Specimens cooled in air expanded on keeping. The chief problem in connexion with the constancy of volume of tool steels is to avoid expansion. It is least for water-hardened steels, and may be eliminated by repeated heating and cooling through an appropriate temperature cycle.
T. H. Burnham.

Specific etching agent for silicon in iron. P. Oberhoffer (Stahl u. Eisen, 1926, **46**, 1191—1192).—0·1*N*-iodine solution diluted to 1 pt. in 10 with alcohol is a specific etching reagent for silicon in iron–oxygen, iron–carbon, and iron–carbon–manganese alloys. The silicon-rich areas are shown up as light spots on a dark ground. Evidence for silicon segregation in certain iron–oxygen alloys deoxidised by silicon is obtained by use of the reagent.
L. M. Clark.

Nature of low-tungsten tool steels. M. A. Grossman and E. C. Bain (Trans. Amer. Soc. Steel Treat., 1926, **9**, 259—276).—An oil-hardening 2½% tungsten steel contracted on tempering up to 175°, expanded up to 260°, and then contracted continuously. A water-hardening steel contracted continuously on tempering unless the hardening temperature was 1000° or above. The oil-hardened steel consisted of austenite and martensite throughout; the water-hardened steel was troostitic at the centre. The expansion was due to the transformation of austenite, of which there were two forms, a rather unstable austenite decomposing at 260° and a small amount of a stable austenite decomposing at 600°. The oil-hardened steel showed secondary hardness at low drawing temperatures and a slight hardening at 600°. Impact tests showed an initial toughening on tempering, an embrittling effect at 260°, then pronounced toughening. The treatment recommended for the best combination of hardness and toughness is to quench at 900° and temper at 175°.
T. H. Burnham.

Corrosion and rusting of steel and iron. R. Girard (Rev. Mét., 1926, **23**, 361—367, 407—417).—In acid solutions of very low concentration the behaviour of iron and steel is similar, but with higher acid concentrations polished iron is attacked rather more than steel. The hydrogen potential at which corrosion commences is greater in weak acids than in strong acids. In regions of constant attack the nature of the acid has little influence, but at places of accelerated attack the corrosion becomes greater with the degree of dissociation of the acid. The conditions governing the formation and existence of the hydroxide film, which commences to be precipitated in acid solutions when the hydrogen potential is 3·5, determine the rate of corrosion. In sodium chloride solutions, iron and steel corrode in the

same manner in the absence of oxygen, but when oxygen is present steel is corroded locally and deeply and the corrosion product is not very adherent, whilst iron is uniformly coated with corrosion product which exerts a protective action. M. COOK.

Recent examinations of steel and iron specimens exposed at Colombo, Halifax, Plymouth, and Auckland. P. M. CROSTHWAITE (Deterioration of Structures in Sea-water. Sixth Interim Report, Comm. Inst. Civ. Eng., Dept. Sci. Ind. Res., 1926, 6—8).—Aerial corrosion seems to be more pronounced at Colombo and Plymouth than at Auckland or Halifax. Submerged and half-tide specimens are in general deteriorating more rapidly than aerial specimens. The most resistant specimens seem to be those of "Galahad" non-corrosive steel, nickel steels, and cast iron of various grades. B. W. CLARKE.

Solubility of silica and other rock-forming substances during cyanidation. C. J. BROOKES (Bull. Inst. Min. Met., Sept., 1926, 11 pp.).—During the cyaniding of the oxidised portion of a gold–silver ore in Sumatra small quantities of alumina and silica were dissolved from the clayey matter present. During the subsequent treatment in the zinc-boxes much trouble was caused by the deposition of a gelatinous precipitate containing silica, alumina, zinc oxide, and lime; the rate of precipitation increased with the richness of the solution in gold and silver, so that it appeared to be due to the coagulating action of the zinc–precious metal couple. Preliminary treatment of the solution with lime water or lead acetate resulted in partial alleviation of the trouble, but, owing to the slow rate of settling of the flocculent precipitate formed, was impracticable. Addition of lime to the ore during grinding overcame the difficulty to a great extent but prevented an efficient separation of sand and slime in the classifier. When the ore was classified without addition of lime, and a good layer of lime then spread on the sand leaching vats, the liquor first passing through was relatively free from silica, but, as the lime became used up, impurities dissolved and subsequently yielded flocculent precipitates when mixed with the first leach liquors. Addition of 1 lb. of ammonium chloride per ton to the leach liquor before use prevented any precipitate from forming on mixing the various effluents. A. R. POWELL.

Production of pure aluminium from insoluble aluminous materials such as clay or alunite. J. YAMAZAKI (J. Soc. Chem. Ind. Japan, 1926, 29, 154—160).—The raw materials used in the investigation were Japanese clay and Korean alunite. By igniting the clay at 700—800° and the alunite at 550°, over 95% of the alumina is rendered soluble in sulphuric acid of *d* 1·69. The clear solution thus obtained contains sulphates of aluminium, iron, and other metals. A sufficient amount of ammonium sulphate is added to convert the whole of the aluminium sulphate into alum, and the solution is allowed to crystallise; the iron salt remains in the solution. By the direct action of dry ammonia gas at 75°, the alum gives aluminium hydroxide and ammonium sulphate; the hydroxide thus prepared is in a compact granular form, easily filtered and washed, and can be purified by washing with water. By drying and igniting the hydroxide, alumina of the following composition is obtained: 99·3% Al_2O_3, 0·111% Fe_2O_3, 0·104% SiO_2, CaO and MgO trace, and 0·4% H_2O. Using this product, metallic aluminium of the following composition is produced: 98·90% Al, 0·76% Si, and 0·15% Fe. The experiments were made on the semi-industrial scale. The cost of production of the alumina on this scale is about £12 14s. per ton and that of metallic aluminium about £87 17s. per ton. K. KASHIMA.

Solidification of aluminium containing iron. G. MASING and O. DAHL (Wiss. Veröff. Siemens-Konz., 1926, 5, 152—159).—Aluminium alloys containing more than 3% Fe expand on solidification and nodules of metal are extruded on the surface. This is not affected by the silicon content and is due to the evolution of gas. The effect is most marked when the alloys are melted in hydrogen and cast in sand. Alloys of high density are obtained with either very rapid or very slow cooling (cf. Archbutt, B., 1925, 286). The eutectic lies at about 2·5% Fe, and the results are explained by assuming that the compound $FeAl_3$, in the liquid state, absorbs gas which is released on solidifying. C. J. SMITHELLS.

Protection of aluminium and its alloys against corrosion by anodic oxidation. G. D. BENGOUGH and H. SUTTON (Brit. Assoc., Aug., 1926; Engineering, 1926, 122, 274—277).—A highly tenacious, thin, glassy film of oxide is produced on aluminium, duralumin, zinc–aluminium alloys, silicon–aluminium alloys containing up to 8·7% Si, and copper–aluminium alloys containing less than 5% Cu by anodic oxidation in 3% chromic acid solution as free as possible from sulphuric acid. The voltage used is raised slowly to 40 volts during 15 min., kept constant for 35 min., raised to 50 volts during 5 min., and finally kept constant for a further 5 min. The coatings thus obtained are highly resistant to sea-water, and break down only after constant wetting and drying; this may be prevented, however, by dipping the treated metal into molten lanoline, or by coating it with paint or varnish. The oxide coating may be dyed any colour by immersing it in a suitable dye-bath, and a glossy coat subsequently obtained by varnishing. The process is applicable to fashioned articles of aluminium, except those having parts of other metals, such as brass or iron. A description of a small-scale and a large-scale experimental plant is given, the cost of operating which is calculated to be less than 2d. per sq. ft. of metal treated. A. R. POWELL.

Special Alpax alloys. M. PETIT (Rev. Mét., 1926, 23, 418—431, 465—484).—In ordinary Alpax (aluminium–silicon) alloys refined or modified by the addition of sodium, the eutectic structure is rendered finer and the strength and hardness are raised as the freezing rate is increased. The optimum quantity of sodium is 0·5%, and is added to the bath at 775°, and the metal cast at 675°. No metal additions to the bath, other than sodium and potassium, have been found capable of refining the alloy. The addition of other metals to Alpax in amounts up to 5% does not very materially improve the mechanical properties. The special metals can be added either before or after the refining process.

The mechanical properties of special Alpax alloys containing copper, magnesium, copper–magnesium, or magnesium–zinc are appreciably influenced by heat treatment. M. COOK.

Electrical resistivity of aluminium–calcium alloys. J. D. EDWARDS and C. S. TAYLOR (Trans. Amer. Electrochem. Soc., 1926, **50**, 33—39. Advance copy).—Addition of pure calcium (99·8%) to pure aluminium (99·94%) increases the resistivity proportionately from 2·70 microhms/cm. to 3·54 microhms/cm. for cast alloys containing 3% Ca and to 3·32 microhms/cm. for similar cold-rolled alloys. The lower resistivity of cold-rolled alloys is due to the breaking up of the eutectic surrounding the grain boundaries. The density of calcium–aluminium alloys containing up to 3% Ca is proportional to the composition. A. R. POWELL.

Phase equilibria of sulphates. II. [Roasting of lead ores.] E. JÄNECKE (Z. anorg. Chem., 1926, **155**, 291—297).—Polemical against Schenck (B., 1926, 589). R. CUTHILL.

Standardisation of microscopical examination of Muntz metal alloys. R. S. PRATT (Min. and Met., 1926, 7, 374—375).—A method of reporting on Muntz metal structures is described which consists of a comparison of the structure in respect of grain size and phase distribution with four standard structures, sketches of which are given. M. COOK.

Manufacture of mixed tin [tin–lead alloy]. R. THEWS (Chem.-Ztg., 1926, **50**, 611—613).—The preparation of mixed tin from scrap bearing metal by the old process, in which copper and antimony are removed by liquation, and zinc is eliminated by burning out, offers no difficulties from the metallurgical standpoint, but economically it is not a success, except for small amounts of material. By the modern method larger quantities of bearing metal are melted down by heating to 800—900° in a specially constructed furnace, the zinc being burnt out, assisted by a secondary air blast. The charge is slowly cooled by a blast of cold air until a thin crust of solid forms, which contains the greater part of the copper and antimony. This crust may be skimmed off and the remainder cooled nearly to the solidification point of the eutectic tin–lead alloy, which is theoretically 182°, but in practice is 183—185°; the liquid alloy is then tapped off. Instead of skimming an alternative method is to cool to the eutectic point, tap off the lead–tin alloy, and then reheat the solid metal left behind, and fractionally melt it, the last fraction being "Glanz" metal containing some 3% of lead. The molten material should be held at the eutectic point for at least 45 min.; in practice, however, some lead–tin alloy is always lost by solidification during the tapping process. W. G. CAREY.

Combustibility of coke and direct reduction in the blast furnace. W. W. HOLLINGS (J. Iron and Steel Inst., 1926, **113**, 285—294).—See B., 1926, 490.

Heterogeneity of steel ingots. Report of subcommittee (J. Iron and Steel Inst., 1926, **113**, 39—176).—See B., 1926, 490.

Distribution of silicates in steel ingots. J. H. S. DICKENSON (J. Iron and Steel Inst., 1926, **113**, 177—211).—See B., 1926, 491.

Ghost lines and the banded structure of rolled and forged mild steels. J. H. WHITELEY (J. Iron and Steel Inst., 1926, **113**, 213—218).—See B., 1926, 491.

Hardness of carbon steels at high temperatures. I. G. SLATER and T. H. TURNER (J. Iron and Steel Inst., 1926, **113**, 295—306).—See B., 1926, 491.

Hardening and tempering of high-speed steel. A. R. PAGE (J. Iron and Steel Inst., 1926, **113**, 307—333).—See B., 1926, 492.

Ratio of tensile strength of steel to Brinell hardness number. R. H. GREAVES and J. A. JONES (J. Iron and Steel Inst., 1926, **113**, 335—353).—See B., 1926, 491.

Effects of arsenic on steel. A. E. CAMERON and G. B. WATERHOUSE (J. Iron and Steel Inst., 1926, **113**, 355—374).—See B., 1926, 491.

Determination of phosphorus in steels containing tungsten. T. E. ROONEY and L. M. CLARK (J. Iron and Steel Inst., 1926, **113**, 457—466).—See B., 1926, 493.

Granulation of slags and metals. B. BOGITCH (Rev. Mét., 1926, **23**, 443—445).—See B., 1926, 547.

PATENTS.

Manufacture of steel. E. BOSSHARDT (E.P. 254,673, 6.11.25. Conv., 4.7.25).—A low-carbon steel possessing a yield limit about 25% greater than that of steel of similar carbon content manufactured by ordinary processes is made by maintaining the temperature at 1800—2000° above the bath in the melting chamber in which there is a reduced pressure. The molten metal is intensely deoxidised and freed from gases by the suction action. As well as ordinary additions, 2% of silicon is added to the bath. The process effects a practically complete removal of sulphur and phosphorus and the product contains 0·05—0·2% C, up to 0·8% Mn, and up to 2% Si. The addition of silicon may be replaced wholly or in part by such metals as tungsten or nickel. M. COOK.

Removal of iron from materials containing it. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 256,428, 3.10.25).—Iron is removed almost quantitatively from materials such as bauxite, titaniferous iron sand, or pyrites, by passing over the reduced material a stream of carbon monoxide at an elevated pressure and temperature, at such a speed that the iron carbonyl formed is rapidly removed. Reduction is carried out by previous treatment with a reducing gas or with carbon monoxide, alone or in admixture with other reducing gases. The carbonyl is decomposed by heat into iron of high purity, and carbon monoxide which is used again in the process. L. M. CLARK.

Briquetting ore concentrates, especially iron ore. V. C. OESTLUND (E.P. 256,838, 3.12.25. Conv., 14.10.25).—A binding material for briquetting ore concentrates consists of the caramel obtained by heating molasses or other waste sugar solution at 200—220°, with or without the addition of water and other agglutinants such as dextrin or starch. A. R. POWELL.

Welding rod and process of welding [steel]. G. E. DOAN (U.S.P. 1,595,143, 10.8.26. Appl., 10.10.21).

—Ductile arc-welded joints between steel articles are formed by melting an iron rod containing up to 3% Mn and 15% C on to the weld metal so that a predetermined quantity of manganese is introduced into the joint.

A. R. POWELL.

Producing gray iron castings. D. H. MELOCHE, Assr. to E. HOLLEY (U.S.P. 1,595,353, 10.8.26. Appl., 31.1.24. Renewed 30.11.25).—Grey iron is cast in a heated metal mould protected by an insulating refractory lining and a superimposed coating of lampblack. The casting is finally heated between 800° and 870° until casting strains are relieved without destruction of the characteristic structure of the iron. L. M. CLARK.

Low-carbon iron–chromium alloys. B. D. SAKLATWALLA (U.S.P. 1,596,999, 24.8.26. Appl., 11.3.22). —Iron–chromium alloys are obtained by smelting in a combustion furnace chrome ore and silicon together with lime and fluorspar in amounts sufficient to render the slag fluid at the working temperature. M. COOK.

Refining silicon-containing iron–chromium alloy. B. D. SAKLATWALLA (U.S.P. 1,597,000, 24.8.26. Appl., 11.3.22).—Iron–chromium alloy containing silicon is refined by smelting it with a mixture containing calcium carbonate, calcium oxide, and a small amount of a nitrate. M. COOK.

Alloy [chrome] steel. B. D. SAKLATWALLA (U.S.P. 1,597,001, 24.8.26. Appl., 7.7.23).—A charge consisting of scrap steel, a silicon reducing agent, and an unreduced chromium compound in sufficient quantities to provide all or most of the iron and chromium in the steel is melted, the chromium being simultaneously reduced from its compound with the formation of chrome steel.

M. COOK.

Decomposition of alloys rich in iron. H. CROTOGINO (G.P. 431,801, 18.6.24).—The finely divided alloys, *e.g.*, furnace sows, are treated with air in the presence of salt solutions containing preferably magnesium salts, and the ferric hydroxide formed is separated from the residual metals by flotation. L. A. COLES.

Dissociating [separating the constituents of bearing-]metal alloys. M. and L. MEYER, Assrs. to HÜTTENWERKE TEMPELHOF A. MEYER (E.P. 241,224, 10.10.25. Conv., 11.10.24).—Tin–lead–copper–antimony bearing-metal is melted in a reverberatory furnace having a rectangular or circular cross-section, and allowed to cool slowly to 182°, at which temperature the lower portion of the metal consisting of the tin–lead eutectic containing about 3% Sb and 0·1% Cu is tapped off, leaving a copper–antimony alloy containing any excess of lead or tin above that required to form the eutectic. The process is adapted to the preparation of a bearing-metal free or nearly free from lead from a leady alloy; this is effected by adding to the charge an excess of tin or tin alloy sufficient to convert all the lead into the eutectic and to leave the necessary tin in the bearing-metal.

A. R. POWELL.

Coating metals. G. A. MEKER (E.P. 245,746, 4.12.25. Conv., 8.1.25. Addn. to 211,122, B., 1925, 212).—A substance such as ammonium chloride or zinc chloride, capable of generating volatile chlorides, is added to the iron–aluminium alloy powder and articles to be coated are heated in the mixture to a temperature between 700° and 1000°. M. COOK.

Metal-coated articles and process of making. S. OTIS and W. T. HERREN, Assrs. to NAT. BOILER WASHING CO. OF ILLINOIS (U.S.P. 1,596,300, 17.8.26. Appl., 11.11.25).—Iron articles are first coated with a film of tin and cadmium by electrolysis, and subsequently coated with lead by any suitable process.

A. R. POWELL.

Manufacture of metals of the cerium group of rare earth metals in the form of thin regular pieces. WESTINGHOUSE LAMP CO., Assees., of J. W. MARDEN and M. N. RICH (E.P. 246,860, 29.1.26. Conv., 29.1.25).—Misch metal or similar alloys of the cerium group of rare earth metals may be extruded in the form of thin wire or ribbon from a press in which the metal is heated to a temperature just below its m.p., *e.g.*, 500—600°. The receptacle into which the metal is extruded is filled with an inert gas, *e.g.*, carbon dioxide, or with an oil. The thin wire or strip thus produced is suitable for use as a "cleaning up" agent in exhausting wireless valves and the like. A. R. POWELL.

Electrolytic manufacture of aluminium. H. DOLTER (E.P. 251,641, 28.4.26 Conv., 4.5.25).—In the ordinary method for the manufacture of aluminium by electrolysis of alumina in a molten fluoride bath, aluminium carbide is added in the proportion of 1 mol. of carbide to 2 mols. of alumina. Alternatively, the anode is made of aluminium carbide or of a compressed and heated mixture of carbon and aluminium carbide. The process gives a yield of double the usual quantity of aluminium for the expenditure of the same amount of electrical energy. A. R. POWELL.

Manufacture of alloys. INTERNAT. NICKEL CO., Assees. of W. A. MUDGE (E.P. 253,879, 21.5.26. Conv., 16.6.25).—The preparation of aluminium–nickel–copper alloys is most advantageously carried out by addition of an alloy containing 35—85% Al, the remainder being nickel or copper or both these elements, to a molten bath of nickel and copper. A small amount of magnesium may be used in the preliminary alloy as a deoxidiser. The product is improved particularly in its hot forging and rolling properties. Improvement is also attained by the use of a refractory containing a small percentage only of silicon. L. M. CLARK.

Aluminium–silicon alloy. A. PACZ, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,595,218—9, 10.8.26. Appl., [A] 27.2 and [B] 3.4.22).—An aluminium–silicon alloy containing 2·5—15% Si contains in addition (A) a small proportion of manganese, or (B) cobalt in an amount not exceeding that of the silicon. A. R. POWELL.

Aluminium alloy. A PACZ, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,596,020, 17.8.26. Appl., 19.4.24). —Silicon–aluminium alloys containing 3—15% Si are melted below a cover of an alkali metal compound containing oxygen and capable of being reduced by some of the constituents of the alloy. The metal is cast while it still contains sufficient of the alkali metal to effect an improvement in its physical properties.

A. R. POWELL.

Improving zinc–aluminium alloys. T. Goldschmidt A.-G. (G.P. 431,985, 16.5.22).—Castings of zinc–aluminium alloys containing the compound Al_2Zn_3 in harmful proportions are homogenised by heating above the decomposition temperature of the compound, or by slowly cooling the fused alloy with or without subsequent quenching, and then heating to a temperature slightly below the decomposition temperature of the compound, until formation of the ductile crystalline variety begins. The addition of elements such as copper, cerium, magnesium, beryllium, titanium, manganese, and chromium is advantageous. L. A. Coles.

Heat treatment and concentration of copper ores. J. C. Moulden, B. Taplin, and Metals Production, Ltd. (E.P. 255,961, 2.5.25).—The copper obtained on the outside of the ore particles by the treatment described in E.P. 250,991 (B., 1926, 590), is separated by froth flotation. Particles of gangue large enough to interfere with the flotation of copper are removed by screening or by water-classification, or the heat-treated material may be ground to reduce the coarse particles to the required fineness. M. Cook.

Manufacture of copper alloys. M. G. Corson (formerly Korsunsky) (E.P. 256,457, 21.12.25).—The addition of 0·1—1·5% of silicon and 0·3—3·0% of iron to copper followed by heat treatment at 950° gives a homogeneous solid solution which is preserved by quenching. A further short treatment at 550—625° increases the Brinell hardness from 50—60 to 90—100, whilst the tensile strength becomes 55,000—63,000 lb. per sq. in., and the elastic limit is over 25,000 lb. per sq. in., with an elongation of 20—35% on 2 in. The specific resistance is about 3·2 microhms. By the addition of 0·1—0·35% of silicon with 0·3—2% of chromium and subsequent heat treatment at 900—950°, followed by quenching and reheating to 450—550°, a similar but rather greater improvement in properties is obtained. The practice may be extended advantageously to alloys of copper containing zinc or tin or other metals. L. M. Clark.

Treatment of ores for recovery of titanium. A. W. Gregory (E.P. 256,836, 30.11.25).—Titanium ores are heated with a mixture of barium sulphate, coal, and a little sodium carbonate, whereby the barium is converted chiefly into barium oxide and the iron of the ore into ferrous sulphide. Subsequent leaching with water and 5% hydrochloric acid in succession removes first the barium salt, then the ferrous sulphide, leaving a titaniferous residue from which the titanium may be readily extracted by heating with concentrated sulphuric acid. A. R. Powell.

Rotary furnace [for roasting zinc blende]. F. E. J. Enke (E.P. 256,861, 30.1.26).—A mechanically-operated, rotary tubular furnace for roasting zinc blende or similar material has a number of roasting chambers arranged parallel to the axis of the furnace. These are interconnected at the ends in such a way that the material to be roasted takes a zig-zag path and the heat generated in the pre-roasting stage is available for the final roasting. The charge may also be passed spirally towards the centre of the furnace or, alternatively, may travel from the centre of the furnace towards the periphery. L. M. Clark.

Separating cobalt from nickel. W. J. Harshaw, Assr. to Harshaw, Fuller & Goodwin Co. (U.S.P. 1,596,253, 17.8.26. Appl., 26.9.24).—A solution of cobalt and nickel salts is treated with a soluble lead salt and then electrolysed, whereby the lead peroxide formed on the anode reacts with the cobalt salt to form cobaltic hydroxide and regenerate the lead salt. A. R. Powell.

Recovery of vanadium. F. F. Frick, Assr. to Anaconda Copper Mining Co. (U.S.P. 1,596,483, 17.8.26. Appl., 16.10.25).—The acid extract of a vanadium-bearing material is evaporated to dryness and the residue mixed with a further quantity of the acid solution and evaporation repeated. A. R. Powell.

Die-casting metal [alloy]. W. M. Peirce and E. A. Anderson, Assrs. to New Jersey Zinc Co. (U.S.P. 1,596,761, 17.8.26. Appl., 11.5.25).—A zinc-base alloy for die-casting contains not less than 85% Zn together with 0·01—0·3% Mg. A. R. Powell.

Recovery of tin from ores, residues, and the like. Soc. d'Électrochimie, d'Électrométallurgie et des Aciéries Électriques d'Ugine (Addn. 30,428, 22.4.25, to F.P. 585,942; cf. E.P. 217,900, B., 1925, 926).—The tin and the addition metal specified in the chief patent are withdrawn from different openings in the smelting furnace arranged according to the densities of the metals. The opening from which the tin is withdrawn is preferably provided with a siphon so as to allow the metal to cool sufficiently to prevent loss by volatilisation. A. R. Powell.

Recovery of tin from tin-plate scrap. H. F. Guillet (Austr. P. 103,475, 26.3.24. Conv., 6.11.23).—Tinned iron scrap is treated with an alkaline hypochlorite solution under such conditions that the final liquor consists of a saturated solution of an alkali chloride containing at least 15 g. of tin per litre as an alkali stannate. This solution is then electrolysed at 50° between insoluble anodes to regenerate the hypochlorite solution for use again in the process and to obtain the greater part of the tin content as pure metal. A. R. Powell.

Enrichment of ores containing limestone. I. Bachilov (F.P. 604,240, 7.8.25).—The finely-ground ore is heated to convert the limestone into calcium oxide and the product is leached with a hot solution of ammonium chloride, whereby the lime dissolves with the formation of calcium chloride and the evolution of ammonia. The latter, together with the carbon dioxide formed in the first stage of the process, is passed into the calcium chloride solution from leaching a previous batch in order to regenerate ammonium chloride and obtain pure calcium carbonate. A. R. Powell.

Production of very finely-divided copper powder. E. Seyfferth (G.P. 431,437, 12.7.25).—Copper formate is heated to 400° in a retort through which a stream of indifferent gas is passed or in which a vacuum may be maintained. A. R. Powell.

Magnesium alloys for hot forging. I. G. Farbenind. A.-G., Assees. of W. Schmidt (G.P. 431,450, 7.1.25).—Magnesium–silicon alloys of the type claimed in G.P. 392,022 (B., 1924, 602) may be worked into sheet and wire, and forged at 400—480°. Other metals may be added to the alloys, provided that they do not lower the

solidus point below 480°. The silicon–magnesium alloys are much more readily worked than magnesium alloys containing zinc and aluminium. A. R. Powell.

See also pages 857, **Heating blast for furnaces** (E.P. 255,281); **Rotary furnaces** (E.P. 256,158). 862, **Gaseous fuel for metal cutting** (E.P. 255,250); **Purifying blast-furnace gases** (E.P. 255,344). 886, **Electric melting furnace** (U.S.P. 1,596,582).

XI.—ELECTROTECHNICS.

See also pages 860, **Transformer oils** (Von der Heyden and Typke); **Increase in viscosity of oils subjected to silent discharge** (Becker). 873, **Preparation of potassium hydroxide** (Vernitz). 874, **Carbide furnace balance** (Baumann); **Manufacture of carbon disulphide** (Rabe). 878, **Electrical resistivity of ceramic materials** (King). 882, **Protecting aluminium and its alloys against corrosion** (Bengough and Sutton). 883, **Electrical resistivity of aluminium–calcium alloys** (Edwards and Taylor). 888, **Use of ozone in drying insulating varnishes** (Spence and Cochran).

Patents.

Electric [induction] furnace. J. A. Seede, Assr. to Gen. Electric Co. (U.S.P. 1,595,967, 10.8.26. Appl., 13.2.25).—In an electric induction furnace a looped channel communicates with a crucible forming a reservoir for the charge, and a number of primary windings are inductively associated with the channel for the purpose of inducing heating currents in the conducting charge contained in the channel. These primary windings are arranged so that oppositely-directed electromagnetic repulsive forces are exerted by the respective windings upon the portion of charge in the channel, whereby circulation of the molten charge between the reservoir and the channel is produced. J. S. G. Thomas.

Induction furnace. Furnace crucible. M. Unger, Assr. to Gen. Electric Co. (U.S.P. 1,595,968—9, 10.8.26. Appl., 18.4.22. [A] Renewed 16.6.26).—(A) An induction furnace comprises a primary, a furnace body shaped to form a reservoir, and a secondary channel of uniform cross-sectional area symmetrically surrounding the primary and communicating with the reservoir at regions radially spaced at different distances from the primary, so that a unidirectional flow of charge between the channel and the reservoir is produced. (B) An induction furnace crucible comprises separable refractory members, which, when in engagement, provide a hollow annular body having inlet and outlet openings. J. S. G. Thomas.

[Operating an] induction furnace. J. M. Weed, Assr. to Gen. Electric Co. (U.S.P. 1,595,970, 10.8.26. Appl., 29.12.12).—An induction furnace in which the charge forms a closed secondary is operated by inducing an electric heating current in the secondary by an alternating electromagnetic field, which is applied so that a difference of fluid pressure is produced between different regions of the secondary, whereby unidirectional flow of the charge through the secondary and return flow through a region external to the secondary is effected. J. S. G. Thomas.

Induction furnace. G. Faccioli, Assr. to Gen. Electric Co. (U.S.P. 1,595,971, 10.8.26. Appl., 5.6.22).—Circulation or interchange of a conducting fluid charge between a reservoir and a communicating, elongated, looped channel of an induction furnace is effected by exerting electromagnetic repulsion upon the charge outwards from a central region, whereby the charge is caused to flow from the channel into the reservoir at regions axially spaced apart, a return flow from the reservoir into the channel occurring at an intermediate region. J. S. G. Thomas.

Electric melting furnace. J. R. Coe, Assr. to Amer. Brass Co. (U.S.P. 1,596,582, 17.8.26. Appl., 18.10.21).—A two-part channel box is secured to a container for molten metal. Between the two parts of the channel box is clamped a lining made up of sections of refractory material, the adjacent inner faces of which provide registering surfaces forming a channel, the ends of which open into the container. Yielding material is interposed between the outer faces of the lining sections and the two parts of the box. L. M. Clark.

Electrodes for electric furnaces. Gebr. Siemens & Co. (G.P. 429,388, 13.6.25).—Cheap and moderately strong electrodes for electric furnaces are manufactured by using milk of lime as binding medium. J. S. G. Thomas.

Precipitating suspended particles from gases or insulating liquids by means of electric fields. Siemens-Schuckertwerke Ges.m.b.H., Assees. of M. Schenkel, H. Mayer, and C. Hahn (G.P. 431,216, 2.6.22).—Electrically neutral or almost neutral suspended particles are passed through a non-homogeneous field formed between two conducting electrodes of opposite polarity, which are connected with an external source of alternating current, the potential being so low that no corona effect or discharge occurs between the electrodes. The spatial distribution and the dielectric constant of the suspended particles may be increased by the introduction of water vapour. The radius of the rod-shaped electrodes employed and their distance apart are in the ratio 0·5 : 1 or 1·1. These cylindrical electrodes are arranged between and within external irregularly corrugated iron sheets, the corrugations being arranged so that both sides of the sheets can be used as electrodes. If desired, rods of circular or elliptical cross-section may be used as precipitating electrodes between lamellar discharge electrodes. J. S. G. Thomas.

Depositing hafnium and zirconium upon incandescent [electric lamp] filaments. N. V. Philips Gloeilampenfabrieken (F.P. 604,391, 9.10.25. Conv., 21.7.25).—Tungsten filaments are heated electrically to about 1450° in an atmosphere of the iodide of hafnium or zirconium. The metal precipitated upon the filament is then deposited upon a second incandescent filament heated to about 1600—1750° in a chamber containing pure iodine. Filaments so prepared can be drawn through very fine dies. J. S. G. Thomas.

See also pages 862, **Purifying blast-furnace gases** (E.P. 255,344). 876, **Electrolytic production of perborates** (G.P. 431,075). 880, **Manufacture of fused cement** (F.P. 604,916). 884, **Metal-coated articles** (U.S.P. 1,596,300). 885, **Separating cobalt from nickel** (U.S.P. 1,596,253); **Recovering tin from tin-plate** (Austr.P. 103,475). 889, **Manufacture of carbon black** (U.S.P. 1,596,070); **Iron oxide pigments** (U.S.P. 1,596,363).

XII.—FATS; OILS; WAXES.

Artificial beef tallow. II. Relation between properties of the soap and the degree of hydrogenation of the oils used. M. Hirose (J. Soc. Chem. Ind. Japan, 1926, 29, 203—212; cf. *ibid*, 1925, 1101).—The surface tension and lathering power of various soaps prepared from hydrogenated soya bean oils were studied for the purpose of comparing the oils with beef tallow. Hydrogenated soya bean oil having an iodine value of 60—69 is the best substitute for beef tallow for the manufacture of soap, although it differs somewhat in composition. The drop number and lathering power of the soap solutions vary with the progress of hydrogenation of the fatty oils used. Drop number and specific volume of lather diminish during the first stage of hydrogenation, then rise gradually to a maximum when the oil has iodine value 60—69, beyond which they again gradually fall until the iodine value of the oil becomes zero. *iso*Oleic acid formed during the hydrogenation has no injurious effect on the properties of the soap. Soaps made from unsaturated fatty acids show a fall of drop number with rise of temperature, whereas those made from saturated acids of high m.p. show the reverse effect, the maximum being generally at the temperature at which the solution becomes quite transparent. K. Kashima.

Oil of *Salvia spinosa* from Cyrenaica. S. Berlingozzi and G. Di Mase (Rend. Accad. Sci. Fis. Mat. [Napoli], 1925, [3], 31, 156—157; Chem. Zentr., 1926, II, 1107).—Seeds of a *Salvia spinosa*, L., from Cyrenaica, which resemble closely the seeds of *Salvia sclarea* (Boll. chim. farm., 63, 721), were dried, pulverised, and extracted in a Soxhlet with a mixture of ether and light petroleum, yielding 9·43% of water and 19·65% of oil. The oil is a fairly mobile, clear, yellowish-green liquid of not unpleasant taste and faintly aromatic odour, d^{15} 0·9298, f.p. below —15°, n_D^{15} 1·4799, m.p. of fatty acids 5—9°, f.p. of fatty acids 3—5°, acid value 1·66, saponif. value 193, Hehner value 94·22, Reichert value 1·3, "relative iodine value" 159·2, "absolute iodine value" 203·6. In the elaidin test the oil gave a yellowish-brown semi-fluid mass. Treatment of the fatty acids with bromine in ether in presence of acetic acid gave a white precipitate, m.p. about 178°, containing 41% Br. As a drying oil the oil is better than that from *S. sclarea*. A. Davidson.

Composition of drying oils, in relation to primary and secondary bromine or iodine values. W. Vaubel (Farben-Ztg., 1926, 31, 2771—2775).—The existence of two bromine values of an oil—"primary" obtained by direct progressive absorption, and "secondary" by use of excess of bromine—is due to the different rates of absorption by acidic components of varying degrees of unsaturation. Linolenic and linoleic acids brominate rapidly, but oleic acid shows no primary absorption. β-Linoleic (*iso*linoleic) acid absorbs two atoms of bromine quickly, but the remaining two are only slowly absorbed, and are thus registered in the secondary bromine value. The primary and secondary iodine values (calculated from the corresponding bromine values of a drying or semi-drying oil) may be used in conjunction with the hexabromide value to determine the content of the oil in acids having one, two, and three double linkings respectively. The maximum iodine value ever recorded for a particular oil is regarded as corresponding to the original composition of the oil, but slow conversion of linolenic acid into β-linoleic acid accounts for the lower values usually obtained for the oils. From the difference between the secondary iodine value and the highest iodine value observed in practice, conclusions may be drawn as to the changes that have occurred on keeping. The compositions of sunflower, soya bean, poppyseed, rapeseed, hempseed, arachis, walnut, linseed, tung, and whale oils are deduced, and the accepted values for some of these oils are discussed in view of the above considerations. S. S. Woolf.

Increase in viscosity of oils subjected to a silent discharge. Becker.—See II.

Polymerisation in drying of oils. (1) Eibner and Munzert. (2) Auer.—See XIII.

Turbidity tests on butter fat and its substitutes. Leduc.—See XIX.

Miscibility tests in detection of adulterated butter. Atkinson.—See XIX.

Patents.

Prevention of rancidity in vegetable oils used in plastic compositions for coating fabric, paper, and the like. G. E. Scharff, and Nobel's Explosives Co. (E.P. 256,654, 6.2.25).—The vegetable oil used in the manufacture of artificial leather or other material made by coating fabric with a composition of nitrocellulose, camphor, and oil, is treated with 2% of finely-ground antimony sulphide or other metal sulphide, which delays the development of a rancid odour. Sulphides of zinc or lead give less favourable results than sulphides of antimony or arsenic. W. G. Carey.

Separation of fatty acids from glycerides. A. Eisenstein (E.P. 257,170, 16.2.26).—Separation of fatty acids is effected by preferential adsorption by soaps rather than neutral oils, the soap being obtained by the usual soap-boiling process, or any oil-insoluble compound of a metal with a fatty or resin acid, or by neutralising a part of the free fatty acids with soda lye. The soaps, with the adsorbed fatty acids, are removed from the glycerides, and are washed with a salt solution specifically lighter than the soap to be removed, and the soaps are precipitated by the dilute salt solutions, setting free any neutral oil adhering to or included in the soap. W. G. Carey.

Production of substances resembling fatty acids [oleine substitute]. A. Riebeck'sche Montanwerke A.-G. (G.P. 429,272, 22.10.21. Addn. to 361,734).—The liquid resin ("talloil") obtained as by-product in the manufacture of cellulose by the soda (sulphate) process, is hydrogenated only to such an extent that it is rendered odourless. L. A. Coles.

Bleaching and refining curd soap. W. Saechtling (E.P. 243,333, 7.11.25. Conv., 24.11.24).—The finished soap, after graining, is treated first with a predetermined amount of hyposulphite solution at 60—70°, stirred well for about ¼ hr., and then treated with an oxidising agent, such as a persulphate, percarbonate, or persilicate. W. G. Carey.

Production of washing and bleaching agents. CHEM. WERKE HERKULES G.M.B.H., Assees. of V. SCHOLZ (G.P. 429,855, 23.9.22).—The material consists of mixtures of peroxides or ozonides of unsaturated organic acids, such as unsaturated resin acids or fatty acids, or of the natural or artificial esters of these acids, with alkalis or substances capable of forming alkalis, such as peroxides or perborates, with or without the addition of compounds such as ammonium salts. The stable polymerised peroxides of natural or artificial esters of unsaturated fatty acids may also be used as oxygen-yielding agents. For example, a detergent yielding 10—20% of active oxygen is obtained by the use of alkali peroxides, linoxyn, and sodium carbonate.

L. A. COLES.

Filtering apparatus (E.P. 256,124).—See I.

Lubricants (U.S.P. 1,594,762).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Report on paint experiments. F. E. WENTWORTH-SHEILDS (Deterioration of Structures in Sea-water. Sixth Interim Report, Comm. Inst. Civ. Eng., Dept. Sci. Ind. Res., 1926, 14—19).—It was noticed that plates of the "B" series (see Fifth Interim Report, 21—31) showed signs of deterioration and rusting a few days after being set up at the half-tide position. After two months the plates painted with linseed-oil paints were in general corroded and rusty over a considerable part of the surface, with considerable peeling off of the paint even where two coats had been applied. Neutralised coal tar, applied direct to the metal or over a coat of red lead–linseed oil paint, seemed to have prevented deterioration up to the time of examination. A bituminous solution of Manjack bitumen in heavy coal-tar naphtha gave quite good protection, but one coat of asphaltum had very little protective action. Galvanised plates were not attacked.

B. W. CLARKE.

"Oxyns." III. Occurrence and detection of polymerisation in the drying of oils. A. EIBNER and H. MUNZERT (Chem. Umschau, 1926, **33**, 188—194, 201—208, 213—216; cf. B., 1925, 679, 930).—The earlier theories of polymerisation are reviewed, special consideration being given to the formation of 4-carbon rings, interaction of peroxide groups with double linkings to form 1—4 dioxan rings, and combination of peroxide groups with one another. These are applied graphically to the possible intra- and extra-molecular polymerisations of typical unsaturated mixed glycerides. Since mol. wt. determinations of oils will not reveal intramolecular polymerisation which does not necessarily lead to an increase in molecule size, more trustworthy information is obtained by saponification of the glycerides and examination of the liberated acids. "Film acids" were isolated from oxyns of linseed and tung oils and of the "stand oils" derived therefrom, and their iodine values, neutralisation values, saponif. values, and mol. wts. by Rast's method were determined. From the numerous values so obtained and the observed facts that oil films are more readily saponified than the original oils, while the corresponding stand-oils are much more resistant to saponification, the inference is drawn that the extra-molecular polymerisation that occurs in heat-thickened oils is absent in the normal oil-drying process, in which auto-oxidation and possible intramolecular polymerisation are postulated as the chief factors. Although the explanation of the whole range of oil-drying phenomena on the lines of changes in degree of dispersion not involving polymerisation is adversely criticised, such changes are regarded as necessary adjuncts to the previous conceptions. The transformation of α-elæostearin into the isomeric crystalline β-modification, which subsequently polymerises, is regarded as analogous to the drying of thickened oils, and not as a part of the normal drying of tung oil. The discussion is extended to such oils as poppyseed oil, soya bean oil, etc. which can yield stand-oils of satisfactory drying properties, although the non-thickened oils dry to poor films that soften spontaneously. Sources of error in mol. wt. determinations receive attention at some length.

S. S. WOOLF.

Polymerisation in the drying process and thickening of fatty oils. L. AUER (Chem. Umschau, 1926, **33**, 216—226).—Previous investigations on the polymerisation of fatty oils are reviewed, references to the original literature being freely quoted. Determinations of changes in mol. wt., iodine value, viscosity, and refractive index, which have been widely instanced as evidence for true polymerisation, are shown to be untrustworthy owing to the intervention of colloid phenomena. The behaviour of styrene in yielding a vitreous modification—metastyrene—(quite distinct from the true dipolymeride, distyrene) is declared to be analogous to the thickening and gelatinising of oils, coagulation of lyophilic isocolloids explaining the so-called polymerisation effects.

S. S. WOOLF.

Utilisation of ozone in the drying of insulating varnishes. L. V. SPENCE and P. B. COCHRAN (Trans. Amer. Electrochem. Soc., 1926, **50**, 15—31. Advance copy).—By stoving oil-varnishes in an oven through which ozonised air is passing, the drying time is considerably reduced. Since the accelerated oxidation causes the film to dry and wrinkle on the surface while the varnish remains soft below, it is necessary to have a period of preliminary air-stoving, varying from 4 to 15 min. at 110°, according to the concentration of ozone used, but drying is still accomplished in half the time of the normal air-stoving process. The insulating properties of films of five typical insulating varnishes (two containing copal, two asphalt, and one ester gum as the resin component), when stoved in ozonised air, were in no way inferior to air-stoved films of equal thickness, while equal resistance to moisture, heat, and oxidation, and the absence of increase in the developed acidity were also demonstrated. Oil-proof films, the properties of which depend on the degree of oxidation of the varnish, may readily be obtained by the ozone process, but their elasticity is impaired.

S. S. WOOLF.

Utility of the Storch–Morawski [Liebermann] reaction in the testing of paints and varnishes. E. STOCK (Farben-Ztg., 1926, **31**, 2777).—The procedure adopted by the German Railways Central Board (EZA) for the detection of rosin (colophony) in paints and varnishes is as follows:—3 drops of the sample are

dissolved in 2—3 c.c. of cold acetic anhydride. The filtered solution is mixed with 2 drops of sulphuric acid (d 1·5). Products that yield a coloration as deep as a 0·001N-solution of potassium permanganate are rejected. The author tested a series of common paint raw materials by this method and found no rosin indicated. The rejection of positively-reacting Albertol varnishes is criticised in view of the modifying treatment undergone by any rosin present in the manufacture of the artificial resin. S. S. Woolf.

Patents.

Preparation of [titanium oxide] pigments and paints. C. Weizmann and J. Blumenfeld (E.P. 256,302, 22.4.25).—The incorporation with titanium oxide or other relatively inert pigments of small proportions of metal peroxides, per-salts, etc. that are capable of reacting chemically with oil-paint media, either in an oxidising or a neutralising sense, improves the hardness and weather-resistance of paints containing the mixture. Zinc hydroxide, zinc carbonate, barium peroxide, etc. are typical substances to be used, with or without zinc oxide, which has previously been used alone as an adjunct to inert pigments. S. S. Woolf.

Manufacture of carbon black. W. O. Snelling (U.S.P. 1,596,070, 17.8.26. Appl., 23.4.25).—Hydrocarbon gases are decomposed by passing them through a thin electrically-heated membrane. S. S. Woolf.

Manufacture of [iron] oxide pigments. J. R. MacMillan, Assr. to Niagara Pigment Corp. (U.S.P. 1,596,363, 17.8.26. Appl., 27.8.25).—Chlorine, obtained by the electrolysis of brine, is passed down a tower packed with iron and sprayed with water, producing a solution of ferrous chloride, which is treated with the liquor containing caustic soda from the electrolytic cells. The precipitate is oxidised after separation from the mother liquor, which is returned for renewed electrolysis. S. S. Woolf.

White pigment. Soc. Gén. Fabr. Couleurs et Prod. Chim. (F.P. 604,494, 2.1.25).—Metallic zinc as strips or cuttings is exposed on wooden trestles to the action of acetic acid and carbon dioxide (combustion gases which have been used to vaporise acetic acid). The zinc carbonate formed is washed off the unchanged metal and ground, moist or dry, with oils. A. Davidson.

Production of golden sulphide of antimony. P. J. F. Souviron (F.P. 605,401, 21.10.25).—Golden sulphide of antimony is precipitated from alkali or alkaline-earth salts of thioantimonic acid in the presence of zinc salts, present in the minimum proportion of 1 mol. of zinc salt to 2 mols. of acid. The resulting product is more fiery and more refractory than the product precipitated without addition of zinc salt. If zinc sulphate be added to the sulphantimonate of an alkaline earth, the precipitate also contains the sulphate of the alkaline earth. J. S. G. Thomas.

Extraction of the manganese content of ochres, earths, and colours. P. J. F. Souviron (F.P. 606,417, 3.11.25).—The pigment is treated with a solution of ferrous sulphate or other ferrous salt, which reduces the higher manganese oxides and renders them soluble. A. Davidson.

Manufacture of colour lakes. Farbenfabr. vorm. F. Bayer & Co. (Austr. P. 103,470, 2.3.23. Conv., 7.3.22).—As precipitants for basic dyes in the preparation of colour lakes, sulphurisation products of phenols, substituted phenols, or condensation products of phenols with aldehydes are used. The lakes are permanently insoluble in water and fast to lime. A. Davidson.

Synthetic resin product. W. F. Fleet, H. V. Potter, and Damard Lacquer Co., Ltd. (E.P. 256,711, 15.5.25).—Phenol or a homologue thereof, and carbamide or a substitution product are heated under reflux for ½ to 1 hr. with formaldehyde or other aldehyde, the quantity of phenol being from one-third to five times the quantity of carbamide, and the amount of aldehyde solution required varying with the ratio of phenol to carbamide. By boiling in an open pan, the condensation product may be dehydrated to an intermediate viscous form soluble in o-toluidine, pyridine, tetrachloroethane, ethyl lactate, and similar solvents, whilst the intermediate product obtained by dehydration *in vacuo* is soluble in more common solvents, *e.g.*, methyl alcohol, acetone, butyl alcohol, etc. Further heating of either intermediate yields an insoluble, infusible product. S. S. Woolf.

Production of resinous condensation products from polyhydric phenols and formaldehyde. L. Helm (F.P. 602,704, 11.12.24).—Polyhydric phenols are condensed with formaldehyde, with or without addition of catalysts, in the presence of the sodium, calcium, or zinc salts of hydrochloric, salicylic, or benzenesulphonic acids. For example, resorcinol is condensed in the usual manner with formaldehyde (40% solution) in the presence of hydrochloric acid or aniline, as well as of calcium chloride or zinc chloride. The presence of the metal salts specified prevents shrinkage or development of brittleness in coverings or solid plastic masses prepared from the condensation products. Similar results are obtained by adding sodium carbonate, borate, acetate, formate, manganese chloride, free acetic acid or benzenesulphonic acid. The polyhydric phenol and formaldehyde solution may also be mixed, before condensation, with glycerol, phenol, acetone, furfuryl alcohol, oils, xanthates, urea and its condensation products with formaldehyde, mineral or vegetable fillers. W. T. K. Braunholtz.

Manufacture of resinous condensation products from phenols and aldehydes. Progress A.-G. (G.P. 426,866, 20.1.23).—Clear, readily-soluble condensation products are obtained by the action of gaseous aldehydes, diluted with inert gases such as nitrogen or carbon dioxide, upon phenols or solutions containing them. For example, a mixture of acetaldehyde vapour and carbon dioxide is passed at 50° for about 5 hrs. through phenol containing a little dilute hydrochloric acid. L. A. Coles.

Manufacture of pure resinous condensation products from phenols and aldehydes. Bakelite Ges.m.b.H., Assees. of F. Seebach (G.P. 431,514, 18.2.25).—Crude phenol–aldehyde condensation products are dissolved in solutions containing alkalis in quantity insufficient to form resin salts, but sufficient to neutralise free phenols and to prevent hydrolysis of the phenoxides

on dilution with water; organic solvents, such as alcohol, alcohol–ether mixtures, methyl alcohol, and acetone, are added if necessary. The resins are then precipitated by dilution with water or with aqueous solutions of hydrotropic salts, such as sodium salicylate, or alkali or ammonium salts of the higher fatty acids, resin acids, sulphonated fats, or hydroxy- or halogeno-fatty acids.

L. A. COLES.

Manufacture of "resites" by the condensation of phenols and aldehydes. C. KULAS and C. PAULING (G.P. 431,619, 13.10.22. Can. P. 240,145, 18.10.23. Swiss P. 106,557, 28.6.23).—"Resitols" formed by condensing phenols and aldehydes under suitable conditions in the presence of hydroxybenzyl alcohols or dihydroxydiarylmethanes, are converted into "resites" by heating in stages. L. A. COLES.

Preparation of condensation products from phenols and their substitution products. G. BRUNN (Austr. P. 100,564, 30.3.21).—Phenol, or one of its derivatives, is heated with thioformaldehyde, thioacetaldehyde, or thioacetone. The thioformaldehyde may be produced from ammonium hydrosulphide and formaldehyde during the condensation process. Aromatic hydrocarbons and their chloro-, amino-, or sulpho-derivatives, as well as oil of turpentine, may also be condensed with thioaldehydes. The products, resembling resins, waxes, tannin, or rubber, are pale in colour and are fast to light. For example, when crystalline phenol is heated to 100° with thioformaldehyde, with or without a condensing agent, a yellow, resinous product is formed which is dehydrated by heating to 160°. Alternatively, a mixture of phenol, ammonium hydrosulphide, and formaldehyde is gradually heated, during 6 hrs. and under pressure, to about 180°, when the solid resin separates from the liquid and can be worked up in the usual manner.

W. T. K. BRAUNHOLTZ.

Production of coloured, transparent, resinous condensation products from phenols and formaldehyde. RESAN KUNSTHARZERZEUGUNGSGES. M.B.H. (Austr. P. 102,677, 4.11.21).—One mol. of phenol or of one of its homologues is treated with more than 1 mol. of formaldehyde in the presence of a weak organic acid and, after the resin has separated, mono-, di-, or trimethylamine or another volatile base is added to the mixture in sufficient quantity to make it just alkaline. The resin is further mixed, during distillation and while still liquid, with a small quantity of a volatile base. For example, a mixture of 1 mol. of phenol with 1, 2, or 3 mols. of formaldehyde (according to the desired hardness of the product), containing a small quantity of a mono- or poly-basic hydroxycarboxylic acid (*e.g.*, salicylic acid) as condensing agent, is heated under reflux for 8—12 hrs. until the mixture appears milky. Dimethylamine is added, together with a small quantity of 1% aqueous ammonia if required, and the mixture is heated for a further 3—5 min., left to cool, and washed repeatedly with distilled water. The moist mixture is then distilled under diminished pressure until it is quite clear, a small quantity of a volatile base is added, and distillation is continued under diminished pressure until only a trace of liquid distils over. The resin so obtained is hardened at 70—110°, with or without application of pressure. The amber-like products are markedly harder, tougher, faster to light, purer, and more transparent than when 1 mol. each of phenol and formaldehyde is used. They have *d* 1·274—1·287, as against 1·26 and 1·27 respectively for the "Resols" and "Novolaks." The resin obtained from 1 mol. of phenol and 3 mols. of formaldehyde resembles natural amber in becoming electrified when rubbed with a cloth. W. T. K. BRAUNHOLTZ.

Paint and varnish remover containing furfuraldehyde. C. ELLIS, Assr. to CHADELOID CHEMICAL Co. (U.S.P. 1,596,413, 17.8.26. Appl., 2.3.23).—A mixture of furfuraldehyde, monochlorobenzene, and a wax is claimed. S. S. WOOLF.

Material for use in printer's ink (G.P. 426,991).—See III.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Variations of plasticity, nerve, and rate of vulcanisation of raw rubber. W. GREINERT and J. BEHRE (Kautschuk, 1926, 207—209).—"Nerve" is regarded as representing the cohesion of the single rubber particles, whilst plasticity represents the resistance of adjacent particles to relative movement. The former is connected with the surface tension of the dissolved rubber; the latter is measured by a modification of the Marzetti plastometer (B., 1923, 1033 A). Both characteristics are undesirably variable for manufacturing purposes. On account of the use of accelerators of vulcanisation variation in the rate of vulcanisation is now less serious in its results, but it is regarded as still important for the manufacture of cold-vulcanised articles. D. F. TWISS.

Is there a substitute for American carbon black [in rubber mixings]? W. B. WIEGAND (India-rubber J., 1926, 72, 385—388).—Refined clays give rubber "compounds" which tear much more easily than corresponding mixtures containing carbon black. In a comparison of lampblack with carbon black it is found that the latter gives products of higher "tensile product" and greater resistance to abrasion than the former; the advantage is in the same direction, whether the vulcanisation accelerator used is organic or inorganic.

D. F. TWISS.

Furfuraldehyde derivatives as rubber accelerators. J. P. TRICKEY and G. J. LEUCK (Ind. Eng. Chem., 1926, 18, 812—813).—Hydrofurfuramide is about one-third to one-half as effective an accelerator as hexamethylenetetramine and diphenylguanidine. The overcure effect is, however, less, and the cured rubber develops a brownish colour on ageing. The behaviour of furfurin is practically identical with that of hydrofurfuramide. Dithiofuroic acid and its salts are of the ultra-accelerator type, giving cures comparable with those obtained by the use of "tuads." The behaviour of two thioaldehydes of the type $(C_4H_3O\cdot CHS)_x$, and of ethylfurfurylamine and furfurylidene-ethylamine, is also tabulated. W. T. K. BRAUNHOLTZ.

Rubbered raincoats and rubber solutions. W. ESCH (Gummi-Ztg., 1926, 40, 2697—2699).—A discussion of chemical features—mainly undesirable ones—possible in fabric and rubber solutions intended for the manufacture of rubber-proofed coats. D. F. TWISS.

XV.—LEATHER; GLUE.

Processes in chrome tanning. S. HILPERT and E. SCHLUMBERGER (Collegium, 1926, 349—356; cf. B., 1926, 639).—Experiments with clear chromium sulphate solutions, and solutions in which precipitation had commenced, have disproved the old rule that the speed of tanning increases with increasing basicity and diminishing precipitation figure. The speed of tanning is a maximum when the product of the chromium concentration and the hydroxyl-ion concentration is a maximum. A graph shows that 96—100% tannage is obtained in 3 hrs. at p_H 2·3 with solutions of chromium sulphate containing about 2000 mg. Cr per 100 c.c., and at p_H 5·0 with solutions containing about 4 mg. Cr per 100 c.c. There is a direct relationship between the logarithm of the chromium concentration and the p_H value at which 96—100% tannage is effected in 3 hrs. Protein swelling depends on the hydrion concentration. It may be represented as the addition of molecules of water, the number depending on the hydrion concentration. The primary amino-groups form the centre, but since they react most easily with the tannins it follows that the more acid the solution the smaller the speed of tannage. The optimum is therefore realised at the isoelectric point. It diminishes with increasing addition of water. This simple conception agrees with the fact that the reaction between quinone and gelatin diminishes so much with increasing acidity that the dark colour produced by the compound can be used for the colorimetric determination of the hydrogen-ion concentration. The hydrion concentration exerts a far greater influence than other factors, which depend on the chemical constitution, *e.g.*, precipitation figure. D. WOODROFFE.

Biochemical problems in leather manufacture. W. SADIKOV (Collegium, 1926, 356—363).—The preliminary wet work involves enzymic and bacterial actions, which appear to free the collagen from all foreign substances, chiefly proteins, which surround and penetrate it. Collagen is an aggregate of *cyclo*peptide micelles. Gelatinisation is not a simplification of the state of aggregation, but a complication thereof, probably a hydrate condensation, since gelatin behaves differently from collagen in its reaction with enzymes. Hypotheses are put forward to explain the stability of collagen towards trypsin. It is suggested that tanning is the condensation of the opened elementary collagen micelles with the hydroxyl groups of the tannin phenols. D. WOODROFFE.

PATENTS.

Oil tanning. A. ROGERS and B. N. MATHUR (U.S.P. 1,595,872, 10.8.26. Appl., 31.8.25).—Furs and skins are tanned with an alkali-containing soapy paste made from marine animal oils and containing an excess of oil. D. WOODROFFE.

Rendering tanning [quebracho] extracts soluble in cold water. GERB- & FARBSTOFFWERKE H. RENNER & Co. A.-G. (G.P. 426,842, 27.7.23).—Quebracho and similar extracts are heated above 100° in an autoclave, *e.g.*, to 125° under 1½—2 atm. pressure, with sulphonic acids or their salts, until a test portion is readily soluble in cold water. Alternatively, the extracts may be heated under pressure successively with bases such as sodium carbonate or ammonium carbonate, and with sulphonic acids, or in the reverse order. Suitable sulphonates include sodium dicresylmethanedisulphonate, sodium 2-amino-8-hydroxynaphthalene-6-sulphonate, sodium coumaronesulphonate, and naphthalenesulphonic acids. L. A. COLES.

Manufacture of tanning agents. I. G. FARBENIND. A.-G., Assees. of T. MARIAM (G.P. 427,999, 27.11.20. Addn. to 416,277; B., 1926, 24).—Tanning agents are prepared by treating nitronaphthalenes with bisulphites in the presence or absence of sulphites, and subsequently converting the products into their aluminium or chromium compounds. For example, α-nitronaphthalene is heated for several hours at 130—150° with a 10% solution of sodium bisulphite and sodium sulphite, after which concentrated aluminium sulphate solution is added to the product, and the sodium sulphate crystals which separate are removed. L. A. COLES.

Manufacture of tanning agents. I. G. FARBENIND. A.-G. (G.P. 429,179, 12.2.24).—Tanning agents are prepared by the action of nitric acid, nitrous gases, or similar oxidising agents on fossil material of vegetable origin, such as peat, lignite, or coal. After oxidation is complete, iron and dark-coloured organic impurities are precipitated by rendering the solution alkaline, and, after their removal, the solution is rendered faintly acid to Congo-red, or alternatively, the solution is rendered neutral to Congo-red, and the impurities are precipitated by the addition of phosphoric acid or soluble phosphates. The purified solution is concentrated to a suitable strength or evaporated to dryness. L. A. COLES.

Characterising hides and leather. A. GANSSER (G.P. 429,801, 24.6.23).—Leather is characterised, enabling it to be subsequently identified, by the addition of small quantities of nickel or its salts to one of the solutions used for treating the hides, *e.g.*, to the pickling or tanning liquor. The metal is absorbed by the skin, and can readily be detected in the ash of a sample. L. A. COLES.

Preventing rancidity in oils used in plastic compositions (E.P. 256,654).—See XII.

XVI.—AGRICULTURE.

Aluminium and acid soils. J. LINE (J. Agric. Sci., 1926, **16**, 335—364).—Investigation of the conditions for the precipitation of aluminium hydroxide and phosphate shows that the former is precipitated at about p_H 4·0, and the latter between p_H 3·0 and 4·0; aluminium cannot therefore exist as a soluble salt even in acid soils. A very small amount (0·001 to 0·006% of the dry weight of the soils examined) remains in solution, probably as colloidal hydroxide, and can be extracted from acid soils by water. No toxic effect towards barley or other plants due to this aluminium could be detected. On addition of aluminium salts to an acid soil, the aluminium is to a large extent rendered insoluble by chemical action or adsorption, but there may be a considerable increase in the acidity and alteration in the buffer properties of the soil. Plant growth is affected if the p_H reached and maintained is harmful to the particular plant. The effect of aluminium on plant

growth in culture solutions may be due either to precipitation of the phosphate and consequent phosphate starvation, or to increased acidity, the plant being unable to change the reaction of the solution owing to progressive hydrolysis of the salt. The beneficial effects of lime and phosphatic fertilisers in naturally acid soils, or in soils to which aluminium salts have been added, are due to their action in reducing acidity or in supplying plant nutrients, and not to precipitation of soluble aluminium. The conclusion is reached that the " toxic aluminium " theory of acid soils is not tenable.

C. T. GIMINGHAM.

Influence of fertilisers and of micro-organisms on the hydrogen-ion concentration of soils. New method for determining p_H. E. AGHNIDES (Internat. Agrik.-Wiss. Rundschau, 1926, 2, 313—326; Chem. Zentr., 1926, II, 1456; Proc. Internat. Soc. Soil Sci., 1926, **2**, 121—133).—The p_H of soils is influenced by the manuring and there is a parallelism between crop yield and p_H, but the yield finally depends on the richness of the soil in aerobic bacteria. The fertility of a soil is directly proportional to its aerobic bacterial activity and inversely proportional to its anaerobic bacterial activity. For the determination of p_H, a suitable indicator is added to 1 c.c. of soil filtrate and the extent of the colour change is measured by the number of drops of an alkaline reagent (50 c.c. of $0 \cdot 2M$-potassium chloride, 50 c.c. of $0 \cdot 2M$-boric acid, and 43·9 c.c. of $0 \cdot 2M$-sodium hydroxide) required to be added to a standard solution of known p_H to give the same colour. The drop relationship between the indicator and the alkaline reagent being known, the p_H of the soil solution is read on a curve corresponding to the indicator used.

C. T. GIMINGHAM.

Origin and nature of soil organic matter or soil "humus," I. Introductory and historical. S. A. WAKSMAN (Soil Sci., 1926, 22, 123—162).—A historical and critical review of our present knowledge of soil organic matter. G. W. ROBINSON.

Base exchange in soil colloids and the availability of exchangeable calcium in different soils. F. W. PARKER and W. W. PATE (J. Amer. Soc. Agron., 1926, **18**, 470—482).—All of the exchangeable base in a soil appears to be in the colloidal material. There is a good correlation between the SiO_2/R_2O_3 ratio of soil colloids and their content of exchangeable base. Soils saturated with univalent bases have a lower heat of wetting and (except for sodium) absorb less water than soils saturated with bivalent bases. The exchangeable calcium in acid soils is found to have a low availability, and in non-acid soils a high availability. A. A. ELDRIDGE.

Reciprocal repression by calcic and magnesic additions in surface soil. W. H. MACINTIRE (J. Amer. Soc. Agron., 1926, **18**, 482—497).—Additions of calcium depress the solubility of native magnesium in surface soil, and *vice versa*. Hence a deficiency of calcium or magnesium must be corrected by addition of the element needed, supplies of which cannot be derived through interchange under humid conditions. A. A. ELDRIDGE.

Composition of biennial white sweet clover as related to soil enrichment. A. L. WHITING and T. E. RICHMOND (Soil Sci., 1926, 22, 83—95).—During the first year of growth under field conditions, in Illinois, the tops of biennial white sweet clover decrease in nitrogen, phosphorus, sulphur, potassium, calcium, and magnesium as the growing season advances. The changes in the composition of the roots are less definite, but there is a certain increase in the nitrogen content. In the second year, the tops show a progressive decrease in the above-mentioned constituents, whilst the roots show a decrease in nitrogen, phosphorus, and sulphur, an increase in potassium, and, apart from slight initial increases, no very definite changes in calcium and magnesium. The composition of the crop may be considerably affected by dressings of ground limestone and fertilisers supplying phosphorus and potassium. Although ploughing in the crop in the autumn does not add so much plant food material to the soil as in the spring, in both cases considerable benefit to the succeeding crop may be expected.

G. W. ROBINSON.

Fixation of calcium-magnesium from burnt limes, limestone, and dolomite incorporations in two soil zones. W. H. MACINTIRE and W. M. SHAW (Soil Sci., 1926, **22**, 109—121; cf. B., 1926, 799).—Data are given to show the fate of incorporations in upper and lower soil zones of calcium hydroxide, magnesian lime, ground limestone, and ground dolomite, respectively, in quantities equivalent to 3570 lb. of calcium carbonate per 2,000,000 lb. of soil, as accounted for by leaching losses, carbonate residues, and increase in non-carbonate calcium and magnesium. The average fixation in 4 years, *i.e.*, the increase in soil non-carbonate calcium and magnesium, from calcium hydroxide, magnesian lime, and the finer grades of ground limestone and dolomite incorporated in the surface zone, was equivalent to 3074 lb. of calcium carbonate per 2,000,000 lb. of soil. For the coarser grades (10—20-mesh) of limestone and dolomite, the average fixation was only equivalent to 2269 lb. of calcium carbonate. In sub-surface incorporations the calcium hydroxide and magnesian lime incorporations gave comparable results but the finer ground limestone and dolomite incorporations gave greater fixation. The practical conclusions are that the coarser the dressing of limestone or dolomite the deeper should be the incorporation; that dolomite should be ground finer than limestone; that a 20—40-mesh product is equivalent to an average 10-mesh product; and that surface-zone incorporations secure the greatest conservation of calcium and magnesium.

G. W. ROBINSON.

Relative merits of mono-, di-, and tri-calcium phosphates as soil fertilisers. (1) S. L. KLING; (2) G. INGHAM; (3) T. D. HALL; (4) H. O'K. WEBBER; (5) H. H. DODDS; (6) C. A. DAWSON (J. S. Afr. Chem. Inst., 1926, 9, 3—9, 10—15, 16—20, 21—23, 24—25, 26—28).—(1) Evidence is put forward to show that the use of monocalcium phosphate (superphosphate) is uneconomical on South African soils, and that better results are obtained over a period of years by the application of ground rock phosphates, either alone or with dung or sulphur. (2) Superphosphate is considered to be the most generally useful form of phosphate on account of its easy diffusion through the soil and precipitation in a fine state of division. A mixture of a soft rock phosphate with an equal amount of superphosphate, which may

be regarded as precipitated dicalcium phosphate, is almost equal to superphosphate in most cases and superior on sour soils. Finely-ground rock phosphates are of considerable value, given a sufficient rainfall. They are best used on definitely acid soils. (3) A mixture of superphosphate and ground phosphate rock is recommended. (4, 5, 6) Further discussion of the subject.

C. T. GIMINGHAM.

Effect of increasing applications of sodium nitrate on sugar beet. J. SOUČEK (Z. Zuckerind. Czechoslov., 1926, **50**, 419—422, 499—503, 507—514).—In a series of field experiments with sugar beet at 65 centres, applications of sodium nitrate at the rates of 0, 100, 200, 300, and 450 kg. per hectare gave average yields of roots of 329, 354, 370, 384, and 398 kg. per hectare respectively; yields of leaves and number of roots per unit area also increased with increasing doses of nitrate and there was a slight increase in the percentage of sugar and of nitrogen in the roots, and in the purity of the juice. The effect of the added nitrogen on the yield and quality of the roots was specially marked on the heavier soils and on those with a low content of nitrogen. On soils deficient in calcium carbonate, the first and second 100 kg. of nitrate showed the largest effects. C. T. GIMINGHAM.

Physiological balance for alfalfa [lucerne] in solution cultures. S. LOMANITZ (Soil Sci., 1926, **22**, 97—107; cf. B., 1925, 48).—Lucerne was grown in culture solutions having an osmotic pressure of 1 atm. with varying proportions of potassium dihydrogen phosphate, calcium nitrate, and magnesium sulphate. The results for tops, roots, and total yield are set out by means of triangular co-ordinates, so as to show the composition of the solutions giving the highest yields. The highest yield for tops and whole plants was given by the solution having potassium dihydrogen phosphate, calcium nitrate, and magnesium sulphate in partial volume molecular concentrations equal to 0·0045, 0·0090, and 0·0045 respectively. For roots alone the corresponding concentrations were 0·0047, 0·0071, and 0·0071. Solutions of this type (*i.e.*, with total osmotic pressure of 1 atmosphere) gave higher yields than Shive's solution R_5C_2 having a total osmotic pressure of 1·75 atm. Other differences are also noted and discussed. G. W. ROBINSON.

PATENTS.

Manufacture of citrate-soluble phosphate. DISTILLERIE DE RUYSBROECK (F.P. 605,686—7, 4.11.25).—Phosphate rock, bone-ash, or the like is suspended in distillation residues, such as vinasses, and the mixture, if necessary with addition of caustic alkali, or alkali carbonate, sulphate, or chloride, is distilled at a temperature not exceeding 600°. If convenient, steam, hydrogen, oxygen, carbon dioxide, carbon monoxide or the like may be present. The conditions are so arranged that, besides the phosphate, a mixture of equimolecular proportions of sodium and potassium carbonates, which melts at 600°, is finally present. The product is treated with milk of lime, or quick lime, and water is gradually added, whereby monocalcium phosphate is obtained, which is separated from the alkali by washing.

C. T. GIMINGHAM.

Fertiliser and glycerin from vinasses. SOC. DES ÉTABL. BARBET (F.P. 605,825, 30.1.25).—The vinasses, cleared by settling, is treated with steam, and after removal of the alcohol still present, is heated by closing the vessel and admitting steam at about 120°. The mass is then filtered, thus yielding a residue which is used as a fertiliser and a filtrate containing glycerin. The latter is evaporated and then distilled *in vacuo* with addition of petroleum burning oil. C. T. GIMINGHAM.

Fertiliser. I. G. FARBENIND. A.-G., Assees. of R. GRIESSBACH (G.P. 431,585, 14.9.24).—The fertiliser consists of difficultly soluble condensation products obtained from formaldehyde and urea, in the presence of other fertilisers (especially nitrogen compounds), if required. The slight solubility of the material makes it possible to use it under water, *e.g.*, in rice culture.

C. T. GIMINGHAM.

Conversion of calcium nitrate into a readily distributable fertiliser. I. G. FARBENIND. A.-G., Assees. of C. EYER and R. GRIESSBACH (G.P. 431,766, 20.6.25; Addn. to G.P. 423,958; cf. E.P. 246,377; B., 1926, 293). —In the process given in the chief patent, the best results are obtained by using approximately equimolecular proportions of calcium nitrate and urea.

C. T. GIMINGHAM.

Manufacture of solid products containing nicotine. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 249,140, 11.3.26. Conv., 14.3.25).—A solid material containing nicotine comprises magnesium carbonate or oxide, in which high percentages of nicotine are absorbed. The material is compressed into tablets from which, on heating, the entire nicotine content is obtained for insecticidal purposes. C. T. GIMINGHAM.

Colourless material for increasing the adhesiveness of insecticides and fungicides. W. SONNE (F.P. 592,569, 2.2.25).—Tung oil is saponified with sodium hydroxide at 40°, and the hard mass obtained is powdered and mixed with gum arabic or animal glue and sugar; it is then dried and again powdered. Calcium arsenate or copper sulphate is added before hardening takes place. Used as a dust (*e.g.*, against cotton or vine pests), the hygroscopic powder adheres well to plants, and is not washed off by rain; it can also be used as a spray fluid with water. C. T. GIMINGHAM.

Manufacture of fluids for control of pests. I. G. FARBENIND. A.-G., Assees. of K. MARX and K. BRODERSEN (G.P. 430,712, 22.12.22).—Acids and other substances of high molecular weight, obtainable by known methods from lignite and other fossil fuels, are converted into alkali salts and precipitated by metal salt solutions; or are mixed with metalloids or metalloid compounds in alkaline solution and precipitated by strong acids or heavy metal salts. For example, lignite is oxidised with nitric acid, extracted with sodium hydroxide, the acids are reprecipitated with hydrochloric acid, neutralised with sodium carbonate, and the salts dissolved in water. A dilute solution of copper sulphate is then added, causing the formation of a fine brown precipitate containing the copper together with organic acids. The suspension settles much more slowly than ordinary Burgundy mixture. It is used as a spray fluid against vine pests.

Sodium sulphide solution saturated with sulphur is mixed with organic acids obtained from peat; dilute sulphuric acid is added, causing evolution of hydrogen sulphide and precipitation of sulphur in intimate mixture with the organic acids. The product is used against mildews. Insoluble organic compounds, such as hydrocarbons, halogen-, hydroxy-, nitro-, and amino-compounds, can be emulsified by stirring with the acids obtained from lignite and precipitated with sulphuric acid. Soluble barium or calcium salts are recommended for emulsifying basic substances. Such emulsions are used as insecticides. The presence of the acids of high molecular weight in these fluids, besides giving increased fineness of precipitate and a more lasting suspension, improves the adhesive properties when sprayed on to foliage. C. T. Gimingham.

Preparing pyrethrum insecticides. H. Yamamoto and K. Inouye, Assrs. to Y. Nosawa (U.S.P. 1,595,538, 10.8.26. Appl., 2.1.25. Conv., 6.5.24).—An insecticide is prepared by extracting the active principle of the pyrethrum plant in the usual manner, adding a suitable quantity of sodium hydroxide, and distilling, thereby removing the more volatile components. C. T. Gimingham.

Stable emulsions of pyrethrum extract. I. G. Farbenind. A.-G. (G.P. 431,699, 22.2.25).—Sulphonated compounds of high mol. wt., ligninsulphonic acid, humic acid, or their salts and allied substances, are used. For example, pyrethrum extract is worked up in a ball mill to a homogeneous mass with the sodium salt of sulphonated naphthol pitch or of benzylnaphthalenesulphonic acid; kaolin, kieselguhr, or the like may be added. The product is either dried and used in the form of a dust or is made up with water and used as a spray. The insecticidal action of such emulsions suffers no loss on long storage. C. T. Gimingham.

Materials containing finely divided mercury (G.P. 427,116).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Purification of beet diffusion juice [using proteolytic enzymes]. H. Schreiber (Ind. Eng. Chem., 1926, **18**, 860—864).—Addition of 1 part of pepsin to 160,000 to 200,000 parts of diffusion juice (1 lb. to 60—75 tons of roots), previously heated to 52°, was found during about 15 min. to raise the purity 2—3°. A process based on this principle (U.S.P. 1,581,663; B., 1926, 561) applied previous to carbonation, was found to economise in lime, carbon dioxide, water, etc., and to give a better clarified juice and a purer sugar. Syrup prepared by this process is said to be edible, having an agreeable odour and taste. J. P. Ogilvie.

Absorption spectra of refinery products and the decolorising efficiency of bone char. T. B. Wayne (Ind. Eng. Chem., 1926, **18**, 847—854).—No definite assertion can be made to the effect that, in general, bone char shows selectivity for any particular colouring matters. Even in the case of the red colouring matters produced by the destruction of sucrose and invert sugar by lime and heat, the decolorising action and selectivity for any particular type of colouring matter varies with the degree of caramelisation. Bone char, however, evidently has some selective action in the removal of green colouring matter derived from the cane, because of the limited solubility of this class of impurities. But the final residual colouring matter present in highly char-filtered liquors of high purity is of a type giving high absorption for light in the red end of the spectrum. J. P. Ogilvie.

Entrainment phenomena in vacuum pans concentrating sugar solutions. C. F. Bardorf (Canad. Chem. Met., 1926, **10**, 175—177).—Examination of the entrainment solids collected during a series of evaporations of sugar liquor in a vacuum pan of the coil type, with devices for catching the entrainment particles apart from the splashings, showed that the quantities of sucrose lost by entrainment in two experiments were 0·057% and 0·083% of the total sucrose and 0·077% and 0·111% of the total solids. The purity of the sucrose in the entrainment solids was much lower than in the original liquor, and the entrainment solids showed a remarkably high percentage of ash, viz., 14·46%, as compared with 0·7% and 1·4% in the solids of the liquor and syrup respectively. Calculated on the initial weights in the sugar liquor, the percentage losses by entrainment of sucrose, invert sugar, non-sugar solids (organic), and ash were in the ratio 1 : 4 : 8 : 30. Dissociation of mineral and organic salts is regarded as an important factor in entrainment phenomena. F. R. Ennos.

Adsorbent carbons. Honig.—See II.

Effect of application of sodium nitrate on sugar beet. Soucek.—See XVI.

Ensilage of sugar-beet tops. Woodman and Amos.—See XIX.

Patents.

Utilising heat in evaporation processes (E.P. 229,649).—See I.

Filtering apparatus (E.P. 256,124).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Review of industries based on yeast. C. Schweizer (Chim. et Ind., 1926, **16**, 190—199).

Patents.

Preparation of alcohol from sulphite-cellulose waste liquor. E. S. Sandberg and H. A. E. Nilsson (G.P. 430,076, 9.9.19. Conv., 16.10.17 and 10.4.19).—The liquor, evaporated to a suitable concentration and, if necessary, neutralised, is fermented, then concentrated to a strength of about 50% of dry substance suitable for the further working up for organic substances, and the condensate distilled in a column apparatus to separate the alcohol. A. Davidson.

Fertiliser and glycerin from vinasses (F.P. 605,825).—See XVI.

XIX.—FOODS.

Preparation of wheat gliadin. M. J. Blish and R. M. Sandstedt (Cereal Chem., 1926, **3**, 144; Chem. Abstr., 1926, **20**, 2548).—Pure gliadin is obtained by treating crude gluten, which has been dried in a vacuum

at 65—70° and powdered, with 0·01—0·1N-acetic acid, filtering, and adding salt or alkali to the filtrate.

A. A. Eldridge.

"Apparent ropiness" (thread formation) in milk due to surface influence. A. T. R. Mattick (J. Agric. Sci., 1926, **16**, 459—465).—A physical form of "ropiness" in milk is described and shown to be due to the formation of thin films of casein and/or lactalbumin at the milk–air interface. The "ropes" are a form of the "mechanical surface aggregates" of Ramsden and may occur on appropriate surfaces, such as ordinary farm coolers, whenever the rate of flow, the temperature, and acidity conditions are favourable. The phenomenon is of importance in handling dilutions of milk for bacterial enumeration.

C. T. Gimingham.

Chamomile (mayweed) and a taint in milk. F. Procter (J. Agric. Sci., 1926, **16**, 443—450).—Chamomile or mayweed, when present in any quantity in hay fed to cows, gives a strong taint to the milk. The species chiefly responsible is *Anthemis cotula*. Tainted milk is produced 12—36 hrs. after commencement of feeding hay containing chamomile. The taint-producing substance is extracted by light petroleum.

C. T. Gimingham.

Turbidity tests on butter fat and its substitutes. J. A. Leduc (Canad. Chem. Met., 1926, **10**, 178—180).—The turbidity number is determined by measuring the volume of precipitant required to produce turbidity in a clear fat solution at a definite temperature. To 5 c.c. of the fat dissolved in 10 c.c. of n-butyl alcohol at 45°, acetone at room temperature is added from a burette until the turbidity is well marked, the final temperature being not lower than 26°. The water content of the acetone is adjusted so as to give a turbidity number of 22·4 with almond oil, as with this strength of precipitant the turbidity numbers of various fats are fairly widely separated. The reagents are quite stable if kept in stoppered bottles. The adulteration of butter fat with 15% or more of coconut oil is easily detected by this method. The detection of smaller amounts of coconut oil is rendered difficult by the variation in the turbidity number for butter fat from different breeds of cows.

F. R. Ennos.

Miscibility tests in the detection of adulterated butter. H. J. Atkinson (Canad. Chem. Met., 1926, **10**, 181—183).—Turbidity numbers were determined using ethyl acetoacetate as precipitant and benzene or chloroform as solvent, the experiments being performed at room temperature with 2 c.c. of melted fat dissolved in 3 c.c. of solvent. The turbidity numbers obtained in this way were quite distinctive, but whereas with benzene as solvent a mixture of 72% of lard and 28% of coconut oil gave the same figure as butter fat, with chloroform the mixture resembling butter fat had the composition 90% of lard and 10% of coconut oil. By the use of the two solvents the presence of 10% of foreign fat in butter may be detected. The reagents being pure organic liquids, repeated standardisation is not necessary.

F. R. Ennos.

Babcock-Gerber method for determining the fat in ice-cream. H. C. Moore and P. A. Morse (J. Dairy Sci., 1926, **9**, 276—285; Chem. Abstr., 1926, **20**, 2545).—A modified Babcock–Gerber method is preferred to the Troy–Fucoma method in comparison with the Mojonnier method as a standard.

A. A. Eldridge.

Determination of the acidity of highly-coloured fruit-type products. C. H. Badger and J. W. Sale (J. Assoc. Off. Agric. Chem., 1926, **9**, 342—346).—In order to render acidulated solutions, coloured with coal-tar colours, titratable to phenolphthalein, the colour is removed by adding woollen cloth and boiling. The cloth is subsequently extracted with boiling water and the washings are titrated, but it is impossible to remove all the acid. The error is not more than 2% and is frequently less.

C. O. Harvey.

Detection and determination of lactic acid in the presence of other organic acids [*e.g.*, in fruit products]. E. K. Nelson (J. Assoc. Off. Agric. Chem., 1926, **9**, 331—333).—A satisfactory method of determining acid in fruit products utilising the advantages of the Kunz and Phelps–Palmer methods (Z. Nahr. Genussm., 1901, 4, 673; B., 1917, 236) consists of diluting 100 g. of the sample with 100 c.c. of warm water and precipitating the pectin etc. with 200 c.c. of alcohol. After straining through linen, evaporating the filtrate to 50 c.c., and acidifying, the liberated acids are extracted with ether for 20 hrs. The extract is treated with 30 c.c. of water, the ether removed by evaporation, and the solution freed from benzoic acid and volatile acids by extraction with chloroform followed by steam distillation. Evaporation with excess of barium hydroxide to a volume of 20 c.c. followed by passing carbon dioxide yields a neutral solution which is freed from barium citrate, malate, and tartrate by adding 67 c.c. of alcohol, water up to 100 c.c., filtering, and washing with a mixture of alcohol and water (2 : 1). After evaporating the filtrate to dryness, the residue is treated with 10 c.c. of the dilute alcohol and again filtered and the residue slightly washed. After evaporation of the filtrate to dryness, the residue is dissolved in cold water, filtered if necessary, and treated with excess of hot quinine sulphate solution, the weight of barium sulphate obtained multiplied by 0·7711 giving the weight of lactic acid. The quinine lactate is identified by evaporation to dryness *in vacuo*, extraction with alcohol free from chloroform, twice crystallising from absolute ethyl acetate, and determining the melting point (165·5°, decomp.).

C. O. Harvey.

Detection of added pepper shells in pepper. E. R. Smith, S. Alfend, and L. C. Mitchell (J. Assoc. Off. Agric. Chem., 1926, **9**, 333—342).—From values obtained for 45 samples of different varieties of whole pepper, pepper shells, and pepper siftings, it is concluded that, by consideration of values for crude fibre, dextrose, and magnesium oxide, in conjunction with the authentic data given, it is possible to detect the presence of 10% or less of added pepper shells in pepper. The presence of pepper siftings is detected by the abnormally high ash content, other values undergoing no marked changes.

C. O. Harvey.

Ensilage of sugar-beet tops. H. E. Woodman and A. Amos (J. Agric. Sci., 1926, **16**, 406—415).—Silage of good quality can be made from sugar-beet tops

alone or mixed with wheat chaff, or from sugar-beet tops and wet sugar-beet pulp ensiled in alternate layers. Analyses of these materials before and after ensiling are given, and digestion experiments on sheep with silage from a mixture of tops and pulp are discussed. When whole tops are ensiled alone, or with pulp, there is risk of large losses owing to the draining away of juice; this is reduced by addition of wheat chaff or other absorbent material. The silage produced in the experiment with wheat chaff was, however, of poorer quality, the odour indicating the presence of butyric acid, and also of trimethylamine, probably arising from betaine in the sugar beet. C. T. GIMINGHAM.

Digestibility trials with poultry. I. Digestibility of English wheats and of fibre in Sussex ground oats. E. T. HALNAN (J. Agric. Sci., 1926, **16**, 451—458).—Digestibility trials with poultry gave results in close agreement for two varieties of wheat. Crude fibre is digested by poultry with difficulty. Except in regard to fibre and ether extract, poultry digest wheat as efficiently as other farm animals. The grinding of the fibre in the preparation of Sussex ground oats does not improve the digestibility. C. T. GIMINGHAM.

PATENTS.

Making a fruit-juice product. H. V. WELCH, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,594,804, 3.8.26. Appl., 5.2.21).—Fruit juice, *e.g.*, lemon or orange juice, is treated according to its composition with suitable amounts of sugar and an alkali, such as magnesium carbonate or magnesia, to give a solution containing a definite amount of sugar and acid, which is sprayed into a current of hot dry air at a temperature below the softening point of the resulting dry powder, *e.g.*, 38—66°. Control of temperature is especially necessary during precipitation of the powder by the Cottrell process. T. S. WHEELER.

Non-souring milk food. M. P. LONG, Assr. to LACTEIN CO. (U.S.P. 1,595,347, 10.8.26. Appl., 19.9.22).—Skimmed milk is soured and agitated until it contains about 3½% of lactic acid. It is then heated gradually to about 32°, and the whey is drawn off. The solids remaining in the whey are precipitated by adding sour whey and heating to about 82°, and are added to the solids previously obtained. The mixture is agitated for several hours with about 0·1% of commercial sulphuric acid, and is allowed to cure for several days, the whey being withdrawn at intervals. H. HOLMES.

Egg product [emulsifying agent]. A. K. EPSTEIN (U.S.P. 1,595,765—6, 10.8.26. Appl., 5.10.25).—Egg yolk is treated with an edible water-soluble organic compound containing a hydroxyl group, capable of preventing denaturing of egg protein during freezing, and with an edible acid for increasing the water-imbibing capacity sufficiently to retain the fluidity without diminishing the viscosity after freezing and thawing. (A) A condimental and preservative essential oil is added. (B) The mixture is maintained frozen until required for use. H. HOLMES.

Improving the baking properties of flour. MÜHLENCHEMIE G.M.B.H. (G.P. 431,749, 22.1.24).—Perboric acid compounds, alone or mixed with other substances, are added to the flour or dough. L. A. COLES.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Comparison of methods of digitalis standardisation. C. L. WIBLE (Amer. J. Pharm., 1926, **98**, 396—401).—The cat method (Hatcher and Brody, *ibid.*, 1910, **82**, 360) and the official frog method (U.S.P. IX, 606) for measuring the therapeutic efficiency of digitalis preparations gave results agreeing within the limits of biological error. The colorimetric method of Knudson and Dresbach (A., 1922, ii, 882) indicated greater activity for each preparation than the above methods, but the results bore no constant ratio to those of the biological methods. The biological methods are applicable with the same degree of accuracy to *Digitalis lutea* as to *D. purpurea*. E. H. SHARPLES.

Hot water infusion (5%) of digitalis leaves and its long-period valuation. FOCKE (Arch. Exp. Path. Pharm., 1926, **115**, 268—276).—The 5% hot-water infusion of digitalis leaves (directions for the preparation of which are given) is, on qualitative grounds, by long-period valuation, preferable to the alcoholic extract. For the purpose of the long-period test, which takes several days, 0·3% of phenol is added to the infusion as preservative. B. FULLMAN.

Relation of some chemical constituents to the grades of Kentucky tobacco. O. M. SHEDD (Kentucky Agr. Exp. Stat. Bull. 258, 1925, 33—58).—A comparative study of the content of total nitrogen, nicotine, nitrate nitrogen, crude ash, silicon, phosphorus, potassium, calcium, and magnesium found in different grades of "burley" and "dark" tobacco grown in Kentucky in 1920 and 1921. The effect of leaf-spot disease as shown by part of the 1920 "burley" crop was to lower the percentage of nicotine without any corresponding diminution in the total nitrogen. F. R. ENNOS.

Preparation and valuation of oleum hyoscyami. H. VALENTIN (Apoth.-Ztg., 1926, **41**, 752—753; Chem. Zentr., 1926, **97**, II, 1449).—A higher yield of oil is obtained in the extraction by using 10 g. of ammonia instead of 3 g. as in the German official method, and by volatilising the ammonia and alcohol on the water-bath. To obtain a green oil it should be prepared in copper or enamel vessels according to the Swiss directions. The acid value should be under 2. To detect hyoscyamine, 10 g. of oil are shaken vigorously with 10 c.c. of chloroform and 10 c.c. of 3% hydrochloric acid; after keeping, the upper layer is filtered through a talc filter, the filtrate made alkaline with ammonia, and shaken with 10 c.c. of ether. The ether extract is evaporated and the residue mixed with 5 drops of a solution of furfuraldehyde in sulphuric acid. On cautious warming an intense violet colour is formed if hyoscyamine is present. E. H. SHARPLES.

Activity of the active substance of the posterior lobe of the human pituitary gland. W. LAMPE (Arch. Exp. Path. Pharm., 1926, **115**, 277—293).—By the method of Smith and McClosky (treatment with acetone; cf. Reprint 822, Public Health Rep., 1923, 493) dry

preparations were made from individual posterior and intermediate lobes of human pituitary glands. Twenty different preparations were all active, large variations occurring in the content of individual glands in active substance. No general connexion could be found between the amount of active substance and the pathological changes of the organism during life. The antidiuretic action of the substance and its action on plain uterus muscle were parallel, the pressor function method of valuation giving partly divergent results. The divergence may be due to external causes, or possibly to the fact that the antidiuretic action and the action on plain uterus muscle on the one hand, and the effect on the pressor function on the other, are due to substances differing chemically and biologically. B. FULLMAN.

Evaluation of thyroid preparations. A. T. CAMERON and J. CARMICHAEL (Trans. Roy. Soc. Canada, 1926, [iii], **20**, V, 1—17).—Tests of thyroid preparations on rats lead to the equation $y = \log(10x + 1)$, where y is the observed effect and x the thyroid-iodine dose per kg. body-weight. Using the curve satisfying this equation in examining eleven different desiccated thyroid preparations it is concluded that dosage of thyroid should be based on the thyroid-iodine values and not on the thyroid itself. W. ROBSON.

Tetanus toxin and its destruction. G. WESENBERG (Z. angew. Chem., 1926, **39**, 1004—1006).—Crude tetanus toxin was precipitated by addition of ammonium sulphate to filtered cultures of the bacteria in neutral broth media. Measurement of the destructive power of various oxidising agents showed that with Chloramine T, calcium hypochlorite (as caporite), and potassium permanganate, 1 pt. of available oxygen renders ineffective 220, 160, and 125 pts. respectively of toxin. With ammonium persulphate or hydrogen peroxide, 1 pt. of toxin requires 0·75—1·5 pts. of oxygen, whilst peroxydase is without effect. L. M. CLARK.

Condensation of methyl alcohol in the presence of contact substances. H. TROPSCH and A. VON PHILIPPOVICH (Abh. Kennt. Kohle, 1925, **7**, 78—83; Chem. Zentr., 1926, II, 1482—1483).—The catalysts examined were those used in the production of "synthol," with a view to elucidate the mechanism of this reaction. In nearly all cases a small quantity of an oil of terpene-like odour was obtained, and the possibility of the formation of higher-boiling compounds was proved. With sodium methoxide, dimethyl ether was formed and the reaction gas was composed almost exclusively of hydrogen. In all other cases, considerable quantities of methane were produced. With an alkaline iron catalyst a considerable amount of oily products was obtained, proving the production of oils direct from methyl alcohol to be possible. W. T. K. BRAUNHOLTZ.

Separation of paraformaldehyde from gases containing formaldehyde. H. TROPSCH and O. ROELEN (Abh. Kennt. Kohle, 1925, **7**, 175—177; Chem. Zentr., 1926, II, 1400).—Concentrated formaldehyde gas can be passed through glass tubes without depositing paraformaldehyde if all impurities be excluded. Substances such as α-methylnaphthalene, nitrobenzene, pyridine, etc. act catalytically in their tendency to cause polymerisation. E. H. SHARPLES.

Determination of iron in ferrum reductum. A. D. HØRLÜCK (Dansk Tidsk. Farm., 1926, **1**, 37—48).—Christensen's method (A., 1905, ii, 654) in which the powder is shaken with a solution of ferric chloride in an atmosphere of carbon dioxide, is untrustworthy. Even when pure electrolytic iron is used the results are always about 1·5% low, due to the fact that the reaction does not proceed wholly according to the equation, $Fe + 2FeCl_3 = 3FeCl_2$, some hydrogen being always liberated, due to acid formed by hydrolysis. In the case of ferrum reductum this hydrogen also reduces the iron oxide which is invariably present. It is recommended that the material should be dissolved in dilute sulphuric acid, and the total ferrous salt in solution determined with permanganate; if a determination of the actual metallic iron in the powder be required, the mercuric chloride method of Wilner (Farm. Tidskr., 1880, 225) is best. The alternative method of the British Pharmacopœia, based on the reaction, $CuSO_4 + Fe = Cu + FeSO_4$, leads to results which are too high unless the dissolved iron be determined gravimetrically, when the method becomes too tedious for general use. H. F. HARWOOD.

Determining lactic acid in presence of other organic acids. NELSON.—See XIX.

PATENTS.

Manufacture of an ester of 4-hydroxy-*N*-allyl-2 : 6-dimethylpiperidine. H. STAUDINGER (E.P. 251,666, 3.5.26. Conv., 4.5.25. Addn. to 232,207, B., 1925 942.) —The base (7 pts.) is heated with benzoyl chloride (10 pts.) at 120° for a short time. The mass is dissolved in ethyl acetate and ether is added to precipitate the hydrochloride, from the aqueous solution of which sodium carbonate precipitates the free ester base, *4-hydroxy-N-allyl-2 : 6-dimethylpiperidine benzoate*, as an oil, b.p. 195°/12 mm. The *hydrochloride* is crystalline, m.p. 159—161° (sinters at 149°). A. DAVIDSON.

Manufacture of acetic anhydride. H. DREYFUS (E.P. 256,663—4, 9.4.25).—(A) Acetic acid vapour is passed through earthenware or, preferably, copper tubes, maintained at 200—700°, or at higher temperatures, and the gaseous products, immediately they leave the reaction zone, are subjected, preferably under reduced pressure, to fractional condensation in apparatus maintained at a temperature between the boiling point of water and that of acetic anhydride. The tubes may contain catalysts, such as sodium sulphate, calcium sulphate, sodium bisulphate, or sodium pyrosulphate, which may be deposited upon porous material such as pumice or kieselguhr. (B) Oxides or carbonates of metals, the acetates of which yield acetone on decomposition at high temperatures, such as barium carbonate, calcium carbonate, zinc oxide, or tin oxide, or finely divided metals, such as zinc, cadmium, aluminium, iron, or lead, are used as catalysts at a temperature below 500°. L. A. COLES.

Making dihydroxy-carbon compounds. H. ESSEX and A. L. WARD, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,594,608, 3.8.26. Appl., 22.11.19; cf. U.S.P. 1,477,113, B., 1924, 200).—Carbon dioxide is introduced into a mixture of an aqueous solution of an alkali or alkaline-earth hypohalogenite, and a carbon

compound containing at least one double linking, when the hypohalogenous acid liberated by the carbon dioxide combines with the unsaturated compound yielding the corresponding halogenohydrin. When reaction is complete the mixture is heated with or without addition of alkali or alkaline-earth carbonate, whereby the corresponding glycol is obtained in satisfactory yield. The preparation of ethylene glycol from ethylene, phenyl glycol from styrene, glycerol from allyl alcohol, phenylglyceric acid from cinnamic acid, and dihydroxystearic acid from oleic acid, is described. T. S. Wheeler.

Treating hydrocarbon gases. C. Ellis, Assr. to S. B. Hunt (U.S.P. 1,594,823, 3.8.26. Appl., 7.2.22; cf. U.S.P. 1,412,233, B., 1922, 404).—Natural gas, casing-head gas, or the like is passed over a nickel, copper, or brass catalyst at about 500—700° under a pressure of about 0·5 atm. at a rate of 3—40 cub. ft. per hr. per sq. in. of cross-section of the catalyst tube, when partial dehydrogenation of the saturated hydrocarbons present takes place. For example, casing-head gasoline, b.p. about 30°, yields at 720° a gas containing 9·6% of ethylene and 32·8% of total olefines. The gases coming from the reaction tube are passed with agitation into cold concentrated sulphuric acid (*d* 1·8) until the density of the acid has fallen to *d* 1·4, when it is diluted to hydrolyse the alkyl sulphates present to alcohols, which are recovered by distillation in a yield of 1·2 litres per 1000 cub. ft. of gas treated. T. S. Wheeler.

Purification of lactic acid. J. W. Lawrie, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,594,843, 3.8.26. Appl., 19.8.21).—A drum rotates slowly partially immersed in a 30—40% solution of crude lactic acid. The drum is maintained at 232—316° and is mounted in an enclosure in which a good vacuum is maintained. The film taken up by the drum vaporises very rapidly, and pure lactic acid, free from water, is condensed by cooling the vapour while still under reduced pressure. There is no formation of lactide or lactic anhydride. T. S. Wheeler.

Producing organic chlorides. R. Thurm (U.S.P. 1,590,265, 29.6.26. Appl., 20.8.25).—Three vols. of pure dry hydrogen chloride and 1 vol. of pure dry carbon monoxide are compressed to 67—200 atm. and passed at 230—400° over a catalyst formed of a metal oxide or chloride or a mixture of such compounds, *e.g.*, equal parts of granulated nickel oxide, alumina, and copper oxide. A mixture of organic chlorides, mainly chloroform and tetrachloroethylene, is obtained, and is condensed with water from the residual gases. The latter are mixed with a further quantity of hydrogen chloride and carbon monoxide and again passed over the catalyst. T. S. Wheeler.

Making phenylethyl alcohol and like compounds. I. F. Harlow and E. C. Britton, Assrs. to Dow Chemical Co. (U.S.P. 1,591,125, 6.7.26. Appl., 4.2.24; cf. E.P. 122,630, B., 1919, 739 a).—Phenyl magnesium bromide dissolved in benzene is heated in a still by means of steam coils so that the benzene volatilises; the benzene is condensed in a receiver to which ethylene oxide is added and the mixture returned to the still. The ethylene oxide and phenyl magnesium bromide react according to the Grignard reaction and a satisfactory yield of phenylethyl alcohol is obtained on adding a dilute mineral acid to the product. T. S. Wheeler.

Deodorising *iso*propyl alcohol. C. O. Johns, Assr. to Standard Development Co. (U.S.P. 1,593,304, 20.7.26. Appl., 17.8.21).—*iso*Propyl alcohol prepared from oil-refinery gases is boiled for 15—60 min. with 1—5% of charcoal which has been activated by heating to redness *in vacuo*, and after cooling has been saturated with oxygen. The product after distillation is free from odour. T. S. Wheeler.

Chlorination of hydrocarbons. Holzverkohlungs-Ind. A.-G., Assees. of E. Krause and K. Roka (U.S.P. 1,591,984, 13.7.26. Appl., 4.6.24. Conv., 15.11.23).—Methane or other saturated hydrocarbon is passed with chlorine and air at 300—650° over cupric chloride, ferric chloride, calcium chloride, activated carbon, or other catalyst which promotes chlorination rather than oxidation. Chlorination of the hydrocarbon occurs with formation of water in place of hydrogen chloride. The presence of the water inhibits explosion. As an example, if 25 litres of methane, 6 litres of chlorine, and 31 litres of air be passed in 1 hr. at 450—500° through 600 mm. of pumice saturated with a solution of cupric chloride and contained in a tube 30 mm. in diameter, a yield of chloro-derivatives of methane corresponding to 93% of the chlorine is obtained. T. S. Wheeler.

Derivatives of aminobenzoic acids. C. F. Boehringer u. Soehne, Assees. of A. Rothmann (U.S.P. 1,591,403, 6.7.26. Appl., 18.7.22. Conv., 20.7.21).—100 pts. of 2-phenylquinoline-4-carboxylic acid are converted into the corresponding acid chloride with thionyl chloride in benzene and the product is treated with anthranilic acid in benzene. The product *4-anthranilido-2-phenylquinoline*, $Ph{\cdot}C_9H_5N{\cdot}CO{\cdot}NH{\cdot}C_6H_4{\cdot}CO_2H$, m.p. 226—227°, is insoluble in water and is of therapeutic value in the treatment of gout and the like. T. S. Wheeler.

Preparation of chlorides of urethanebenzoic acids [ethyl carboxyphenylcarbamates], and their side-chain homologues. Établ. Poulenc Frères (F.P. 579,203, 1.6.23).—Urethane-carboxylic acids are treated with thionyl chloride or phosphorus trichloride. *p*-Urethanebenzoic acid, m.p. 201—202°, from ethyl chloroformate and sodium *p*-aminobenzoate, when heated to 100° with thionyl chloride until dissolved yields the acid *chloride*, $COCl{\cdot}C_6H_4{\cdot}NH{\cdot}CO_2Et$, which softens without melting at 110°, and cannot be distilled in *vacuo*. The acid *chlorides* of *m*-urethanebenzoic acid (free acid, m.p. 193°), *p*-urethanephenoxyacetic acid, $CO_2H{\cdot}CH_2{\cdot}O{\cdot}C_6H_4{\cdot}NH{\cdot}CO_2Et$, and *p*-urethanephenylacetic acid, $COOH{\cdot}CH_2{\cdot}C_6H_4{\cdot}NH{\cdot}CO_2Et$, m.p., 125—126°, which is obtained from ethyl chloroformate and *p*-aminophenylacetic acid, are described. *p*-Urethanebenzoyl chloride and diethylaminoethyl alcohol form the *hydrochloride* of the *urethane* of *p*-diethylaminoethyl benzoate (*ethyl diethylaminocarbethoxyphenylcarbamate hydrochloride*), $CO_2Et{\cdot}NH{\cdot}C_6H_4{\cdot}CO_2{\cdot}CH_2{\cdot}CH_2{\cdot}NEt_2,HCl$, m.p. 172—173°, which has a pronounced anæsthetic effect on the tongue. The *hydrochloride* of ethyl *p*-urethanebenzoate melts at 125—127°. J. H. S. Davies.

Water-soluble metallic organic compound. M. S. KHARASCH (U.S.P. 1,589,599, 22.6.26. Appl., 24.4.24).—An organometallic compound of the type RMX_n, where R is an organic radical, M is a metal or metalloid such as mercury, arsenic, antimony, or bismuth, which can be linked to carbon to form stable compounds, and X is an inorganic radical, is treated in aqueous solution with a water-soluble organic compound containing the SH-group, *e.g.*, ethylmercaptan or thioglycollic acid, to give water-soluble compounds of the type, $R{\cdot}M{\cdot}S{\cdot}R'_n$, which are of therapeutic value. For example, if phenylmercuric chloride (1 mol.) is added to an aqueous solution of potassium thioglycollate (1·25 mols.), and the solution acidified, *phenylmercurithioglycollic acid*, $Ph{\cdot}Hg{\cdot}S{\cdot}CH_2{\cdot}COOH$, m.p. 164°, separates.
T. S. WHEELER.

Preparation of *N*-aminobenzoyl derivatives of aminoarylarsinic acids. ÉTABL. POULENC FRÈRES (F.P. 583,338, 28.9.23).—The compounds are prepared by reducing nitroacylaminoarylarsinic acids obtained by the action of aromatic nitrocarboxylic acid chlorides on aminoarylarsinic acids, or by the alkaline hydrolysis of urethanes obtained by the action of urethanoarylcarboxylic acid chlorides on aminoarylarsinic acids. The products can be converted into carbamide derivatives by treatment with carbonyl chloride. For example, a mixture of a solution of sodium *p*-aminophenylarsenite in 20% aqueous sodium acetate with an ethereal solution of *p*-urethanobenzoic acid chloride is stirred for 1 hr., yielding *pp'-urethanobenzamidophenylarsinic acid*, $AsO_3H_2{\cdot}C_6H_4{\cdot}NH{\cdot}CO{\cdot}C_6H_4{\cdot}NH{\cdot}CO_2Et$, which on hydrolysis with dilute sodium hydroxide yields *pp'-aminobenzamidophenylarsinic acid*. Methods are also described for preparing 3:3'- and 3:4'-*amino*- and 3:4'-*nitro-benzamido-4-hydroxybenzene-1-arsinic acid*, and *pp'-nitrophenylacetamidophenylarsinic acid*, in which the nitro-group can be reduced to the amino-group by treatment with sodium hyposulphite in the cold.
L. A. COLES.

Preparation of [A] 4-amino-2-bismuthimercaptobenzoic acid, and [B] 4-amino-2-cupromercaptobenzoic acid. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of W. SCHOELLER and E. BORGWARDT (G.P. [A] 426,345, and [B] 426,346, 28.5.24).—4-Amino-2-mercaptobenzoic acid is treated, in alkaline, acid, or neutral solution, with (A) bismuth salts, or (B) cuprous salts. The *bismuth* compound $[NH_2{\cdot}C_6H_3(CO_2H){\cdot}S]_3Bi$, is an orange-yellow insoluble powder. The *cuprous* compound, $NH_2{\cdot}C_6H_3(CO_2H){\cdot}SCu$, is yellow. The respective *sodium* salts are obtained by dissolving the bismuth or cuprous compound in sodium hydroxide solution and precipitating with alcohol. They have therapeutic application.
A. DAVIDSON.

Preparation of ethyl-*sec.*-butylbarbituric acid. M. L. C. ROGER (F.P. 598,072, 20.8.24).—*Ethyl*-sec.-*butylbarbituric acid*, m.p. 165°, is obtained by direct condensation of the disubstituted malonic ester with carbamide; by condensing *ethyl ethyl*-sec.-*butylcyanoacetate*, b.p. 120°/12 mm., with carbamide in presence of sodium ethoxide, and hydrolysing the resulting *ethyl*-sec.-*butyliminobarbituric acid*, m.p. above 240°, with mineral acid; by treating with ethyl iodide the sodium salt of sec.-*butylbarbituric acid*, m.p. 190°, obtained by condensing *diethyl* sec.-*butylmalonate*, b.p. 117°/14 mm., with carbamide in presence of the same condensing agent; or by condensing *ethyl* sec.-*butylcyanoacetate*, b.p. 118°/17 mm., with carbamide and hydrolysing the resulting sec.-*butyliminobarbituric acid*.
J. S. H. DAVIES.

Preparation of γ-amino-ββ-diethylbutan-α-ol. P. BILLON (F.P. 601,968, 8.11.24).—The oxime of ethyl αα-diethylacetoacetate is reduced with sodium and absolute alcohol (cf., A., 1925, i, 120).
L. A. COLES.

Production of materials containing mercury in a finely-divided, stable condition. I. G. FARBENIND. A.-G., Assees. of O. FALEK (G.P. 427,116, 9.11.24).—Suitable amounts of aluminium powder and mercury compounds are ground together to form an amalgam, which is treated with sufficient water to form a powder or a paste. In this way a loose powder containing aluminium hydroxide and finely divided mercury (11—7%) is obtained. In a similar way, from aluminium cuttings, mercuric oxide, and water a uniform grey paste results containing 3—4% of mercury is obtained. The products are used for therapeutic purposes and as insecticides and fungicides.
W. G. CAREY.

Preparation of a mixture of digitalis glucosides. C. MANNICH (G.P. 427,274, 15.8.23).—A solution in dilute alcohol of a digitalis leaf extract which has been mainly freed from "ballast" material (prepared conveniently as in G.P. 383,480, B., 1924, 578) is precipitated with basic lead acetate, the filtrate freed from lead, and the neutral lead-free solution concentrated in a vacuum until the glucosides separate as faintly coloured, rather swollen grains. They are washed with a little water and purified by solution in dilute alcohol and slow dilution of the solution. They are then free from tannoids, chlorophyll, and saponins. Their activity attains that of pure digitoxin. The part of the mixture containing digitoxin is soluble in chloroform. Another part, insoluble in chloroform, can be separated into two further portions by cold absolute alcohol.
A. DAVIDSON.

Preparation of water-soluble protein derivatives. I. G. FARBENIND. A.-G., Assees. of H. WEYLAND (G.P. 427,275, 11.1.25).—Vegetable or animal proteins or materials containing them are treated with alkylene oxides, if necessary under pressure and with moderate heating. The protein is decomposed by the process to a varying extent, and the alkylene oxide becomes firmly attached to the decomposition products. For example, meat meal is heated with ethylene oxide for 5 hrs. under pressure at 100°. The brown alkaline solution is soluble in water, alcohol, and mineral acids, gives a strong precipitate with tannin, mercuric chloride, and phosphotungstic acid, but is no longer precipitated by picric acid. Other examples relate to similar products from casein and propylene oxide, fibrin and ethylene oxide, edestin and ethylene oxide, etc. The products find therapeutic application.
A. DAVIDSON.

Preparation of diphenylsuccinic dinitrile. I. G. FARBENIND. A.-G. (G.P. 427,416, 8.7.24).—To a boiling alcoholic solution of α-phenylcinnamonitrile a hot aqueous solution of potassium cyanide is added slowly. As soon as the solution darkens, a saturated solution of

ammonium or magnesium chloride is added in small portions. After boiling for about ¾ hr., *diphenylsuccinonitrile* separates. A. DAVIDSON.

Separation of nopinene from pinene–nopinene mixtures. G. AUSTERWEIL (G.P. 427,418, 19.11.24).—Mixtures of the two terpenes are systematically extracted with organic solvents diluted with water, the extent of the dilution varying with the temperature used. The nopinene collecting in the washing liquid is separated either by precipitation or by fractional distillation. For example, from French oil of turpentine, by repeated washing with 65—72% alcohol at 15°, a product containing about 80—82% of nopinene (β-pinene) is separated. The solubility of the α-pinene in 72% alcohol is about 2%, that of the nopinene about 9%. In 65% alcohol, the solubilities at 15° are 0·6% and 3·3% respectively. These solubilities are increased by about 30% by using *iso*propyl alcohol, and diminished by about 20% with methyl alcohol. The nopinene may be used for the synthesis of terpin hydrate or borneol. A. DAVIDSON.

Separation of terpene mixtures or of difficultly separable terpene derivatives. G. AUSTERWEIL and L. PEUFAILLIT (G.P. 428,860, 6.9.22).—A membrane is used which has varying permeability for the different components of the mixture, diffusion taking place into solvents. For example, pinene may be separated from camphene by a vulcanised rubber membrane, using methyl or ethyl alcohol or acetone, which does not swell the membrane. A solution, in chloroform or alcohol, of the terpene mixture formed in the oxidation of camphene to camphor, may be separated by a membrane prepared from a common solution of cellulose acetate and nitrate in acetone, using alcohol or chloroform on the other side of the membrane. The procedure can also be used for the separation of valuable constituents of essential oils as well as for the removal of terpenes from essential oils. Since no rise of temperature takes place, the finer odorous properties do not suffer. Technically, besides flat membranes, rubber or cellulose-ester tubes may be used (possibly with reinforcement for thin membranes), inserted concentrically in the pipes of a condenser, but so as to form a quite separate tube system. The condenser jacket contains the solvent at the required temperature and the mixture to be dialysed is circulated through the inner tube. If oil of bergamot is passed through this arrangement, using a rubber tube and alcohol or acetone, almost pure limonene is extracted. Similarly, camphene can be separated from oil of turpentine by 95% alcohol. A. DAVIDSON.

Preparation of a basic compound of tri-iodophenol and bismuth. KNOLL & CO. CHEM. FABR. (G.P. 430,684, 29.5.25).—An alkaline tri-iodophenol solution is treated with the calculated quantity of an acid bismuth nitrate solution. The mixture should be alkaline, and is afterwards acidified, when *bismuth tri-iodophenol*, $(C_6H_2I_3O)_2Bi_3O_2(OH)_3$, a loose yellow powder, is formed. The preparation is applied to the treatment of abscesses, furunculosis, eczema, and suppurating wounds. A. DAVIDSON.

Preparation of 4-hydroxydiphenyl-4′-arsinic acid. C. F. BOEHRINGER & SÖHNE G.M.B.H., Assees. of A. ROTHMANN and H. STEIN (G.P. 429,103, 3.9.24).—4′-Amino-4-hydroxydiphenyl hydrochloride is diazotised with concentrated hydrochloric acid and nitrite, and the diazo-solution run into an aqueous sodium arsenite solution. Frothing takes place, and after warming to 80° to complete the reaction, the solution is partly neutralised with hydrochloric acid, which precipitates dark brown impurities. These are filtered off, the red alkaline filtrate is concentrated with addition of animal charcoal, and the product precipitated by hydrochloric acid. After dissolving again in hot water it is obtained as white flocks. *4-Hydroxydiphenyl-4′-arsinic acid*, $OH{\cdot}C_6H_4{\cdot}C_6H_4{\cdot}AsO_3H_2$, m.p. above 300°, has valuable therapeutic properties. A. DAVIDSON.

Preparation of a derivative of 4-amino-2-auromercaptobenzoic acid stable in aqueous solution. I. G. FARBENIND. A.-G., Assees. of K. STREITWOLF and P. FRITZSCHE (G.P. 429,150, 10.6.22).—The derivative is prepared by the action of formaldehyde-bisulphite on the sodium salt of the acid. The two components are mixed in aqueous solution, the clear solution poured into methyl alcohol, and the yellow precipitate filtered off, washed with methyl alcohol and ether, and dried in a high vacuum. The product is very soluble in water with almost neutral reaction, and the hot solution is stable under nitrogen. The solution can be used directly for injections. No gold is split off by the formaldehyde-bisulphite derivative even on warming. The preparation withstands tropical climates. A. DAVIDSON.

Preparation of a derivative of 4-amino-1-phenyl-2 : 3-dimethyl-5-pyrazolone. K. THOMÄ CHEM. FABR., Assees. of M. GÖTTLER (G.P. 429,219, 27.9.24. Addn. to 409,715, B, 1925, 473).—Benzoylated hydrastinine is used in place of the benzoylated cotarnine specified in the chief patent. *Benzoylhydrastinine-4-amino-1-phenyl-2:3-dimethyl-5-pyrazolone*, m.p. 207—208°, is lemon-yellow, tasteless, and easily split into its components by mineral acids. It is used as a haemostyptic. A. DAVIDSON.

Manufacture of ethylene chloride. I. G. FARBENIND. A.-G., Assees. of O. ERNST and H. WAHL (G.P. 430,539, 21.4.22).—Ethylene is acted on by hydrogen chloride and oxygen in presence of catalysts at high temperatures. The reaction follows the equation: $C_2H_4 + 2HCl + O = C_2H_4Cl_2 + H_2O$. The hydrogen chloride can be applied either as gas or as vaporised aqueous solution, the oxygen in pure condition or as air. The process is a continuous one. The most advantageous catalysts are copper compounds, but gold and such metals as cause formation of chlorine from hydrogen chloride and oxygen are also suitable. For example, a mixture of 100 vols. of air, about 20 vols. of ethylene, and at least 40 vols. of hydrogen chloride is passed through an acid-resisting contact chamber containing lumps of pumice soaked with cupric chloride at 300°. The escaping gas mixture is well cooled and led through ice-water, where water, excess of hydrogen chloride, and most of the ethylene chloride condense. From the residual gases, after washing out the last traces of hydrogen chloride, small amounts of ethylene chloride can be absorbed by active carbon. Besides ethylene chloride only traces of other chlorinated hydrocarbons,

e.g., ethylidene chloride and higher chloro-derivatives of ethane, are formed. A. Davidson.

Purification of the methyl alcohol, higher alcohols, esters and the like, obtained synthetically by treatment of oxides of carbon with hydrogen. I. G. Farbenind. A.-G., Assees. of M. Müller-Cunradi (G.P. 430,623, 15.1.24).—The substances are treated in the hot liquid or gaseous condition, conveniently by distillation, with oxalic acid. Crystalline or anhydrous oxalic acid (2% or less) is added to the liquid before distillation, or the vapour of the liquid is led over a layer of oxalic acid or of material impregnated with oxalic acid. Together with oxalic acid, porous absorption carbon may be used, or the absorption carbon may be impregnated with oxalic acid and added to the methyl alcohol or the vapour of the latter led over the mixture. The foreign odour of the synthetic methyl alcohol is thus removed. Similarly, the odour of the higher alcohols and esters is improved. Acid oxalates may also be used in place of oxalic acid. A. Davidson.

Preparation of unsaturated β-keto-bases. C. Mannich (G.P. 430,683, 23.4.24).—Unsaturated ketones are heated with formaldehyde and salts of secondary aliphatic amines, or cyclic amines of the type of piperidine, in presence or absence of solvents or diluents. For example, an absolute alcoholic solution of diethylamine hydrochloride is treated with benzylideneacetone and paraformaldehyde and the mixture heated for 1 hour under reflux. All volatile matter is distilled off at 40° in a vacuum, the viscous mass stirred several times with ether, and dried, yielding 1-N-*diethylamino*-5-*phenyl*-4-*penten*-3-*one hydrochloride*, m.p. 112·5°. From the aqueous solution alkalis precipitate the free *base* as a clear odourless oil. From piperidine hydrochloride, veratrylideneacetone, and paraformaldehyde is obtained 1-N-*piperidino*-5 : 3′ : 4′-*dimethoxyphenyl*-4-*penten*-3-*one hydrochloride*, yellowish, m.p. 188°. Piperonylideneacetone, diethylamine hydrochloride, and paraformaldehyde yield 1-N-*diethylamino*-5 : 3′ : 4′-*methylenedioxyphenyl*-4-*penten*-3-*one hydrochloride*, yellowish, m.p. 165°. The free *keto-base* from allylacetone, paraformaldehyde, and piperidine hydrochloride is a colourless liquid, b.p. 122—126°/16 mm. From piperidine hydrochloride, paraformaldehyde, and acetone is formed 1-N-*piperidino*-5-*phenyl*-4-*penten*-3-*one hydrochloride*, m.p. 180·5°. Condensation of nitropiperonylideneacetone with piperidine hydrochloride and paraformaldehyde yields 1-N-*piperidino*-5 : 2′ : 3′ : 4′-*nitromethylenedioxyphenyl*-4-*penten*-3-*one hydrochloride*, yellow, m.p. 175—177°; the free *keto-base* forms yellow crystals, m.p. 127°. The hydrochlorides of the keto-bases are stable towards acids. The bases are mostly oily and inclined to resinify. They are valuable intermediates for pharmaceutical products. A. Davidson.

Preparation of carbamic esters. Kalle & Co. A.-G., Assees. of E. Spröngerts (G.P. 430,732, 9.6.23).—Chloral alkoxides are, *e.g.*, condensed with phosgene, in presence of dimethylaniline or quinoline, to the chloral alkoxide carbonyl chlorides, and these are converted into carbamic esters by ammonia or organic bases, such as aniline. Alternatively, alkyl- or aryl-carbonyl chlorides are converted, through the mixed carbonates by treatment with ammonia, into carbamic esters. The following esters are described: *carbamic ester of chloral ethoxide*, m.p. 109°; *phenylcarbamic ester of chloral ethoxide*, m.p. 78°; p-*ethoxyphenylcarbamic ester of chloral ethoxide*, m.p. 88°, from *p*-phenetidine and the chloroformate of chloral ethoxide; *carbamic ester of chloral methoxide*, m.p. 121°; *carbamic ester of chloral propoxide*, m.p. 85°; *carbamic ester of chloral allyl oxide*, m.p. 64°; *carbamic ester of chloral* iso*amyl oxide*, m.p. 70—72°. The products are good hypnotics, are non-irritant, and their hypnotic and toxic doses are widely different. A. Davidson.

Preparation of water-soluble complex antimony compounds of the quinoline series. I. G. Farbenind. A.-G., Assees. of H. Hahl (G.P. 430,883, 29.11.24).—Alkali or alkaline-earth salts of 8-hydroxyquinolinesulphonic acids are treated with oxygen compounds of tervalent antimony. For example, an aqueous solution of sodium 8-hydroxyquinoline-5-sulphonate is heated with antimony sesquioxide till the latter is almost wholly dissolved, then filtered, and the filtrate evaporated. The antimony complex compound is a yellowish-white powder giving a yellow, almost neutral, solution in water. Again, if a neutral aqueous solution of 8-hydroxyquinolinedisulphonic acid (cf. J. pr. Chem., 1890, **41**, 40) is heated for some time with excess of antimony hydroxide, filtered, and the filtrate dropped into alcohol, an antimony complex compound precipitates as a yellow powder giving a faintly acid solution in water. It contains about 9% Sb. From the aqueous solutions of both products alkali sulphide precipitates orange-red antimony sulphide, Sb_2S_3. The aqueous solutions are non-irritant, and have therapeutic application. A. Davidson.

Preparation of triacetamidotriphenylstibine. Chem. Fabr. von Heyden A.-G., Assees. of H. Schmidt (G.P. 431,202, 3.1.22. Addn. to 389,151; B., 1924, 809).—Triacetamidotriphenylstibine is prepared by the process described in the chief patent by heating acetamidophenylstibine oxide with methyl alcohol. The product is applied therapeutically either directly or after such further treatment as removal of the acetyl groups or oxidation of the antimony residue (cf. Schmidt, A., 1922, i, 1203). A. Davidson.

Production of alkaloid preparations from drugs, especially from ergot. Chem. Fabr. "Norgine" V. Stein, and W. Wiechovski (G.P. 431,512, 12.7.22).—Before treating the drugs or their aqueous extracts with alcohol, the active bases are combined with di- or poly-basic acids, and the bases subsequently recovered by precipitating the acids as insoluble salts. For example, a suspension of finely ground *Secale cornutum* in water is allowed to ferment for 2—3 days, basic lead acetate or lead carbonate is added, and lead is precipitated with hydrogen sulphide. After concentration, the filtered solution is extracted with alcohol, the residue after evaporation again extracted with alcohol, and tartaric acid added to a 10—20% alcoholic solution of the extract, until a test portion no longer becomes cloudy on the addition of tartaric acid; the precipitate is extracted with water, and the extract heated successively with *N*-sulphuric acid and a 10% aqueous phosphotungstic acid solution until a filtered test portion no

longer gives turbidity with phosphotungstic acid. The precipitate is decomposed with lead acetate, and the solution, after removal of lead, is concentrated, and the residue dissolved in alcohol and again precipitated with tartaric acid. The product, a white, soluble, amorphous powder, gives the characteristic reactions for ergot extract. Methods are also described for separating active bases from *Capsella bursæ pastoris* and from *Ustilago maidis*, and a previously undescribed *alkaloid* from cinchona bark. L. A. Coles.

Purification of bisalkylxanthens. C. F. Boehringer & Söhne G.m.b.H., Assees of A. Rothmann and H. Stein (G.P. 431,752, 29.1.24).—The compounds are treated with a current of an inert gas, if necessary *in vacuo*. For example, air containing 5—10% of chlorine is passed through an ice-cold potassium ethylxanthate solution until a test portion no longer gives a precipitate with copper sulphate. The precipitate is dissolved in ether and the solution evaporated to dryness, yielding almost odourless bisethylxanthen. Crude bisethylxanthen can be purified by steam distillation *in vacuo* at 80—90°, or by treatment with a current of dry air. The pure products possess anti-parasitic properties, and are of therapeutic value, *e.g.*, in treating scabies and mange in dogs. L. A. Coles.

Solutions for intravenous injection. H. Schirokauer (G.P. 431,847, 14.11.23).—Products yielding stable solutions suitable for intravenous injection in the treatment of bilharzia are prepared by combining potassium antimonyl tartrate with 2-phenylquinoline-4-carboxylic acid or its derivatives, in the presence of excess of an organic base such as hexamethylenetetramine. L. A. Coles.

Catalysing apparatus (U.S.P. 1,580,740).—See I.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Covering power of photographic silver deposits. S. E. Sheppard (Phot. J., 1926, **66**, 470—472).—In measuring covering power, silver is determined by a micro-colorimetric method as colloidal silver sulphide. Emulsions vary greatly in their covering power for the same development conditions, and in the tendency to a real constancy of the "photometric constant." The photometric constant decreases for high densities, even after an initial constancy, but with some emulsions no region of constancy exists, either with varying exposure and constant development, or with constant exposure and varying development. The degree of variability of the photometric constant, or the covering power, is probably a definite emulsion characteristic. A great difference was observed between the covering power of image density, *i.e.*, due to light, and of fog density, viz., fog produced on prolonged development. On sufficiently prolonged development of an unexposed plate, all the silver halide was reduced, but the maximum density for this was only 1·34. A fully exposed area, developed, gave a density approaching 6·5. Covering power of a silver image appears to be determined not only by the original grain size, but also by the number of sensitising nuclei and their conversion into development centres. W. Clark.

XXII.—EXPLOSIVES; MATCHES.

Tracer mixture. L. Hendler, Assr. to U.S. Secretary for War (U.S.P. 1,593,721, 27.7.26. Appl., 22.10.23).—A pyrotechnic composition comprises an alkaline-earth nitrate and carbonate, potassium nitrate, magnesium or aluminium, red lead, and calcium or other resinate to bind the components together and increase the brilliance. E. S. Kreis.

XXIII.—SANITATION; WATER PURIFICATION.

Determination of iodine in drinking water. W. Steffens (Z. angew. Chem., 1926, **39**, 1098—1100).—The residue from the evaporation of 50 litres of water is repeatedly extracted with 96% alcohol, the extract is evaporated to dryness, the residue dissolved in 10 c.c. of water, and the organic matter oxidised by the addition of permanganate. Alternatively the organic matter may be destroyed by evaporation to dryness followed by gentle ignition of the residue. The iodide in solution is decomposed by addition of sodium nitrite or potassium permanganate and sulphuric acid, and the liberated iodine extracted by shaking with carbon disulphide. By the use of the centrifugal microcolorimeter of Fellenberg and only 0·02 c.c. of the disulphide, the presence of very minute traces of iodine is readily detected. If the water contains bromide the carbon disulphide solution is shaken with a dilute solution of potassium thiocyanate added drop by drop until the brown colour disappears, leaving the pure violet colour due to iodine. A. R. Powell.

Patents.

Sewage treatment by activated sludge. K. Imhoff, F. Fries, and F. Sierp (E.P. 256,489, 5.2.26).—A small quantity of air under pressure is supplied to the aeration tank at a place where the ascension of the bubbles through the liquid is opposed to the direction of movement of the liquid by the mechanical agitating device, thus promoting a circuitous flow of the bubbles to the liquid level. H. Holmes.

Apparatus for treating organic residues. L. Boggiano-Pico (U.S.P. 1,595,445, 10.8.26. Appl., 31.10.24. Conv., 7.10.24).—A pit for sewage water is arranged under a tower providing a fermentation chamber, and a partition between the tower and the pit is provided with ports for exhaust of excess liquid from the chamber. Means are provided for introducing the material into the chamber, for removing the treated material from the chamber, for introducing sewage water from the pit into the chamber, and for forcing air through the chamber to support the fermentation. H. Holmes.

Filling for gas protection apparatus. F. Kerschbaum (U.S.P. 1,595,788, 10.8.26. Appl., 13.4.26. Conv., 11.4.25).—One layer for adsorption of irritants and another for adsorption of poisonous gases are used. The relative thicknesses and adsorbing qualities of the layers are such that the layer for adsorption of irritants becomes exhausted before the other layer. H. Holmes.

See also pages 876, **Alkali salts of chloroimidodisulphonic acid** (G.P. 431,116). 893, **Solid products containing nicotine** (E.P. 249,140); **Insecticides and fungicides** (F.P. 592,569); **Fluids for control of pests** (G.P. 430,712). 894, **Pyrethrum insecticides** (U.S.P. 1,595,538); **Emulsions of pyrethrum extract** (G.P. 431,699). 899, **Materials containing finely-divided mercury** (G.P. 427,116).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOVEMBER 12, 1926.

I.—GENERAL; PLANT; MACHINERY.

Theory of fine grinding. II. G. Martin, E. A. Bowes, and J. W. Christelow. **III.** G. Martin, E. A. Bowes, and F. B. Turner (Trans. Ceram. Soc., 1926, **25**, 51—62, 63—81; cf. J.S.C.I., 1926, 160 T).—II.—A method of determining accurately the surface of crushed sand particles consists in exposing the material to the solvent action of hydrofluoric acid under definite conditions of time and temperature, measuring the loss in weight, and equating this against the loss in weight of a specimen of quartz having an accurately measurable surface, such as a cube. The experiments were carried out with 5*N*-acid in considerable excess, a water thermostat being used to maintain a temperature of 25°. The acid and sand, in a bakelite container, were shaken together for one hour by means of special apparatus. The loss of weight of a known surface of quartz under the conditions of these experiments was 0·0000418 g./sq. cm. using *N*-acid. III.—To determine the relation between the work expended and the degree of fineness (surface produced) in grinding sand, a given weight of sand was ground for varying periods in a ball mill. The power consumed was accurately measured by electrical apparatus, and the surface of the crushed sand was determined by the hydrofluoric acid method (see above). The results showed that, in ordinary tube mill grinding, the surface produced is directly proportional to the work done. To avoid the "cushioning" effect of the dust in extremely fine grinding, the dust must be removed in an air stream. The work required in grinding sand to any given degree of fineness with a given grinding medium is found by the formula: $W = B(S_2 - S_1)$ ft.-lb., in which S_2 is the final surface of the powder in sq. ft., S_1 the original surface of the sand in sq. ft., and B a constant peculiar to the grinding medium and the material ground, representing the work required to increase the surface by 1 sq. ft. B varies considerably with the grinding medium used; with 1-in. steel balls in an 18 × 18-in. mill, it is 60·9 ft.-lb. F. Salt.

Determination of the relative surface areas of powdered materials. W. A. Koehler (J. Amer. Ceram. Soc., 1926, 9, 437—443).—Relative surface area measurements were made, on the assumption that, for a given material, the adsorption of thorium-*B* is a function of the surface, regardless of the degree of fineness of the particles. From the amount of thorium-*B* adsorbed, which is determined with an electroscope, the surface area relations were calculated. The thorium-*B* solution was dissolved either in distilled water or in ethyl alcohol, with or without the addition of ammonia. Equal weights of various powdered materials were placed in equal volumes of the radioactive solution, and, after equilibrium had been established, 10 c.c. of the clear supernatant liquid from each sample were evaporated to dryness. The relative radioactivities of the residues were then measured with the electroscope. Since the adsorption does not vary directly with the quantity of adsorbing material present (this relation being expressed approximately by the Freundlich adsorption isotherm), it was necessary to determine adsorption curves for each material. The method is illustrated by reference to quartz sand. For silica and silicon carbide, 2 c.c. of concentrated ammonia solution per 100 c.c. of thorium-*B* solution were required to effect adsorption, whilst with kaolin the results were vitiated owing to the slaking action of aqueous solutions. The use of anhydrous alcohol instead of water still gave erratic results with this material. F. Salt.

Proposed British standard table of wire screens. II. O. C. Ellington (J.S.C.I., 1926, **45**, 353—354 T; cf. B., 1926, 471).—Amended proposals are made for a standard table, in which the screens are designated by whole numbers of ten-thousandths of an inch, expressing with sufficient accuracy the average aperture widths required. It has been confirmed (1) that the nomenclature proposed will present no difficulty to weavers; (2) the use of half-gauge wires is unnecessary and inadvisable; (3) no coarser screens can be included in the proposals, since such are not normally machine-woven; (4) brass and phosphor-bronze wires are to be preferred to those of copper, because of their greater strength, durability, and resistance to corrosion; (5) although the screens proposed will not meet all special and peculiar trade requirements, they are suitable for all normal screening purposes, and form a suitable basis for authoritative standardisation.

Problems in connexion with ancient Egyptian materials.—A. Lucas (Analyst, 1926, **51**, 435—450).—Some of the unsolved problems in connexion with the following ancient Egyptian materials are described:—flint implements, faience, glass, metals, mortar and plaster, oils and fats, pigments and varnish, pottery, resinous materials, bitumen, coloured mummy hair, stone, and textile fabrics. D. G. Hewer.

Thermoregulator. J. Hume (J.S.C.I., 1926, **45**, 246 T).—A regulator, requiring no relay, is made from a steam-pressure gauge, the mechanism of which acts as a switch. Movement is caused by the expansion and contraction of the toluene with which the apparatus is filled.

See also A., Oct., 1001, **Adsorptive power of charcoals** (Surun); **Adsorption of gases by wood charcoal** (Magnus and Kahn); **Wood charcoal as adsorbent for gases** (Magnus).

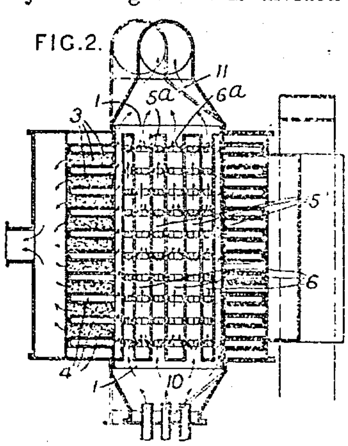
Miscometer. Houston.—See XIX.

Patents.

Steam or other vapour power plants utilising [A] caustic potash, [B] soda, [C] caustic soda, or like auxiliary fluid. E. Koenemann (E.P. 242,979—81, 6.11.25. Conv., [A—B] 14.11.24, [C] 12.11.24).—(A) The bulk of the lye is taken from a mixer at an intermediate pressure and has its pressure raised by a pump, and then passes forward through a heat interchanger to a primary evaporator which is heated from an external source. The more concentrated lye returns through the heat interchanger and a throttle valve to the mixer. The vapour from the primary evaporator does work in an engine and is then sprayed into the mixer. A smaller part of the lye is passed by a circulating pump through the heating coil of a secondary evaporator, and returned to the mixer, also in the form of spray, where its combination with the primary vapour produces heat. Low-pressure vapour from the secondary evaporator does useful work and is condensed. In (B) the mixer is in thermal contact with the secondary evaporator and the lye passing forward through the heat interchanger is raised to a higher pressure than in the primary evaporator, throttle valves being used after the lye has passed the heat interchanger in each direction. In (C) two units, each comprising a mixer and an evaporator in thermal contact, are used, and the flow of lye is from the first mixer through a pump to the second evaporator, to the first evaporator through a throttle to the second mixer, and back to the first mixer. There is also a rapid local forced circulation of lye between the bottom and top of each mixer. Vapour from a power plant enters the first mixer and produces heat by combination with the lye and the heat passes by conduction to the adjoining evaporator. Vapour from the latter does work and passes to the second mixer. Heat from the last produces vapour in the second evaporator, which is also available for work. B. M. Venables.

Devices for drying, roasting, or otherwise treating materials in bulk. O. Dobbelstein (E.P. 250,224, 26.3.26. Conv., 4.4.25. Addn. to 226,543; cf. B., 1925, 655).—A slowly rotating drum is divided into annular

FIG. 2.



compartments used alternately for heating (4) and for containing the material (3). The annuli for heating are provided with radial guiding partitions, alternate sides of which are in communication with passages, 5, for heating gases, or 6 for exhaust gases in the central part of the furnace. The passages 5 are closed at one end of the furnace and the passages 6 at the other.
B. M. Venables.

[Continuous] kiln. H. Freeborough (U.S.P. 1,590,935, 29.6.26. Appl., 7.3.22).—A multi-chamber kiln has fire boxes extending along the sides of each chamber, one wall of each fire box terminating near the top of the kiln. Channel-like members extend across each kiln near the end, and communicate with the space beneath the perforated floor. The channel of one chamber is connected by a passage with the fire box of the next. Additional channels, placed equidistantly between the end channels, also communicate with the space beneath the floor. F. Salt.

Tunnel kiln. W. E. Wilson and H. G. Lykken (U.S.P. 1,590,462, 29.6.26. Appl., 14.6.24).—The cooling zone of a tunnel kiln has a convection space above the trucks carrying the ware. F. Salt.

Pulverising mill. Strachan Tube Mill Co., Assees. of C. B. Strachan (E.P. 252,331, 29.3.26. Conv., 25.5.25).—See U.S.P. 1,583,324; B., 1926, 520.

Production of dry liquefied gases. J. Y. Johnson. From Badische Anilin & Soda Fabrik (E.P. 257,372, 22.6.25).—See F.P. 600,028; B., 1926, 392.

Centrifugal apparatus for dust separation [from gases]. J. Keith & Blackman Co., Ltd., and G. Keith (E.P. 257,301, 27.3.25).

[Centrifugal] apparatus for separating or expelling solid impurities from air, combustion gases, etc. O. Stott (E.P. 257,321, 25.5.25).

Dust collectors or separators and the like. Davidson & Co., Ltd., and J. Whitmore (E.P. 257,403, 14.8.25 and 5.12.25).

Recuperative heat exchange apparatus for air or other media. A. Sandberg (E.P. 245,111, 18.12.25. Conv., 19.12.24).

Woven fabrics for use as air filters. Hoover, Ltd. From Hoover Co. (E.P. 257,484, 14.1.26).

Emulsifying apparatus. J. McGougan and J. Hunter (E.P. 257,704, 4.7.25).

Air seals for furnaces with travelling grates. L. Winter, and Kleb & Bark G.m.b.H. (E.P. 252,201, 17.5.26).

Purification and reclaiming of lubricating oil. Akt. Separator, Assees. of F. R. C. Boyd (E.P. 240,438, 9.9.25. Conv., 26.9.24).

Evaporating and crystallising apparatus (E.P. 252,686).—See XVII.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Change in weight of coal during prolonged oxidation at low temperatures. D. J. W. Kreulen (Fuel, 1926, 5, 345—346).—A Scotch (Wemyss Leven) coal was ground to pass 80 meshes per cm., and 5 g. were heated at 105°; the loss in weight during intervals of time for 4000 hrs. was determined and a circular graph constructed. A sudden loss in weight of 7·92% first

occurs, due to the removal of water, and the subsequent changes, due to oxidation, take place in three distinct periods. In the first there is an increase in the weight of the coal, in the second (968—1275 hrs.) there is an equilibrium between the increase in weight by fixation of oxygen and the loss in weight due to the decomposition of the coal, and in the third period there is a loss in weight. A. C. Monkhouse.

Determination of fineness of coal dust. E. F. Greig (Safety in Mines Research Board, Paper No. 25, 1926, 31 pp.).—The size of particles and their specific surfaces (surface per unit mass of dust particle) may be determined from measurements of linear dimensions (microscopical counting, sieving), by methods based on terminal velocities (sedimentation, elutriation, electrical precipitation), or by empirical methods (bulk-density, heat of wetting, angle of repose, filtering constants). Sieving is considered insufficiently accurate; air-elutriation methods provide means of obtaining grades of dusts of definite ranges of specific surfaces for the purpose of correlating specific surface and degree of inflammability of a dust-cloud; by a combination of elutriation, sedimentation, and microscopic examination it is possible to grade the specific surfaces of dusts. Some of the empirical methods (*e.g.*, percolation through a bed of the material under test) may be useful for rapid determinations and field use. W. T. K. Braunholtz.

Combustion of particles of coal in air. Cenospheres. II. H. E. Newall and F. S. Sinnatt (Fuel, 1926, **5**, 335—339).—Six coal seams were examined in connexion with the formation of cenospheres (cf. B., 1925, 87). Cenospheres were obtained only from the vitrain and clarain of coking seams, and not from durain or non-coking seams. Ignition points were obtained for a number of coal dusts by allowing the coal to fall through a heated tube at 650° and measuring the mean distance fallen before ignition occurred. Individual screened particles from three coals were similarly examined and it was concluded that with a caking coal a cenosphere is first formed, and the films or " windows " then undergo oxidation, leaving a particle consisting of lattices, which subsequently burn. Cenospheres and particles of carbonised durain examined at 600°, 650°, and 700° were found to be more readily inflammable than the original coal particles, due in the case of the cenosphere to the larger surface per unit mass, and with durain to the tarry matter on the surface. A. C. Monkhouse.

Influence of moisture on the process of combustion, especially of coke. P. Oberhoffer and E. Pivovarsky (Stahl u. Eisen, 1926, **46**, 1311—1320).—The effect of moisture in the blast on the process of combustion of coke in an open-shaft furnace has been investigated in a series of tests in which observations have been made of the maximum temperature obtained in the furnace, the thickness of the zones of maximum temperature and of maximum carbon dioxide content, composition of the flue gas, and rate of combustion of the coke. With a blast of relatively low humidity the maximum temperature is higher; the zones of maximum temperature and maximum carbon dioxide content are lower and the carbon monoxide content of the flue gases is greater the higher the moisture content of the air. Above a certain limit of humidity the maximum furnace temperature begins to decrease with an increase in the moisture, until when the air is nearly saturated a rapid fall in the maximum temperature takes place, accompanied by a rapid decrease in the carbon dioxide content of the gases and a corresponding increase in the carbon monoxide content of the flue gas. The introduction of the moisture in the form of spray into the tuyères instead of in the form of vapour in the blast causes a more rapid gasification of the coke and a maximum temperature 50—60° higher to be obtained. Increasing the rate of supply of blast or its temperature up to 400° improves the process of combustion in a manner similar to that obtained by the addition of a little moisture to an almost dry blast. An explanation of the beneficial effect of small quantities of moisture is given, based on a theory of surface adsorption of moisture. A. R. Powell.

Examination and valuation of coking coals. R. Kattwinkel (Fuel, 1926, **5**, 347—355).—A comparison is made with coals from six Westphalian coke-oven plants of the crucible test, the tube-distillation (Bauer) test, and the caking index (Meurice) test as compared with the results obtained in large-scale practice. The Meurice coking test is modified by using a mixture of coal and sand in the proportions of 1 : 10 and a crushing strength apparatus with lead shot in order to apply a gradually increasing load. The experimental methods are found to be suitable for the classification of coals, but the results are not sufficiently comparable with those obtained in practice. The theories of Fischer (B., 1925, 233) correlating the bitumen content with the coking properties of coal are similarly deficient, being based on crucible carbonisation. A. C. Monkhouse.

Thermal decomposition of coal in presence of catalysts and under hydrogen pressure. A. R. Bowen and A. W. Nash (Fuel, 1926, **5**, 361—364).—Arley coal was heated for 2—3 hrs. up to 480° with hydrogen under pressure in an autoclave (B., 1926, 474), in the presence of nickel and iron oxides, stirring being carried out by the admixture of small steel cylinders to the charge. Under comparable conditions 45·64% of oil products was obtained with nickel oxide as compared with 30·95% with iron oxide. The proportion of phenols and bases in the oils obtained with both oxides was approximately the same, and independent of the oil yield; mean values were phenols, 9·21%, bases, 6·41%. The ether-insoluble and chloroform-soluble fractions were less and the light spirit fraction was greater with increased temperature. Iodine values showed the lighter fractions to be not fully saturated. A. C. Monkhouse.

Parr process of low-temperature carbonisation of coal. W. R. Chapman (Fuel, 1926, **5**, 355—361).—The process is primarily designed for the carbonisation of coal of high oxygen content and is carried out in two stages. The coal, ground to ¼-in. size, is fed into a cylindrical rotating retort of 8 in. diam., horizontally mounted and heated externally by flue gases and gas burners. This preliminary carbonisation takes place slowly to 100° and then rapidly to 300°, which is 50° below the temperature of the plastic stage of the coal. The charge (400 lb.) is then transferred from the preheater to a steel vertical retort 12 ft. high and 12—14 in. diam.

maintained at 750—800° and the products of distillation are collected. In this stage, due to an exothermic reaction equivalent to 65 B.Th.U. per lb. of coal, rapid carbonisation occurs, yielding a coke of 3—4% volatile matter suitable for a domestic fuel. The total time of carbonisation is 4 hrs. A. C. MONKHOUSE.

cycloPentanone in the distillation products of lignite. D. VORLÄNDER and W. GÖRNANDT (Z. angew. Chem. 1926, **39**, 1116—1117).—The presence of *cyclo*pentanone (0·1%) in the crude fraction, b.p. 110—140°, obtained in the distillation of lignite has been detected by the formation of the crystalline-liquid anisylidene-*cyclo*pentanone (cf. Vorländer, A, 1921, i, 867) on treatment with anisaldehyde and sodium hydroxide. The ring ketone is more soluble in water than is generally recognised in the literature, and may be extracted by shaking the crude oil with water, and the aqueous distillate and wash liquors are rich in acetone and *cyclo*pentanone. Treatment of the crude oil with sulphuric acid at ordinary temperature or washing with alkalis as employed in the purification of motor spirit, removes 90% of the *cyclo*pentanone. The higher fraction of the oil (b.p. 140—180°) contains a very small quantity of *cyclo*pentanone, and no *cyclo*hexanone was found in either fraction. J. W. BAKER.

Peat. I. Thermal decomposition of peat under reduced pressure. J. REILLY and G. PYNE (Sci. Proc. Roy. Dublin Soc., 1926, **18**, 329—341).—In the rapid distillation of dry peat in a vacuum there are obtained less ammonia, acetic acid, methyl alcohol, and gaseous products and more tar, especially of the higher b.p. or more paraffinic fraction, than is the case in distillation at the ordinary pressure. The difference is probably due to less secondary decomposition of the more complex products. The gases evolved are similar in the two cases. G. M. BENNETT.

Effect of anti-knock materials on spontaneous ignition temperatures of some inflammable liquids. Y. TANAKA and Y. NAGAI (Proc. Imp Acad. Tokyo, 1926, **2**, 221—223).—Small amounts of diethyl selenide, lead tetraethyl, pyridine, or aromatic amines raise the spontaneous ignition temperatures of alcohols very considerably, but have little or no effect on those of hydrocarbons or ethyl ether. R. CUTHILL.

Firedamp explosions: projection of flame. M. J. BURGESS (Safety in Mines Res. Bd., Paper 27, 1926, 14 pp.).—The distance of projection of the flame produced on igniting a mixture of methane and air has been determined under varying conditions. The apparatus consisted of a glass tube, 5·5 cm. in diameter in one series of experiments and 9 cm. in a second series, which was divided into two parts by a brass shutter. One part contained the combustible mixture, which was ignited by a spark between electrodes at the end of the tube remote from the shutter. The latter was opened just before ignition so that the flame was projected into the other part of the tube, the distance of projection being determined by visual observation in a darkened room. The diameter of the aperture through which the flame was projected could be varied. The projection of the flame into air in an unconstricted tube was between five and six times the length of the original column of explosive mixture, mixtures richer in methane giving a longer projection than weak mixtures of corresponding explosive power, owing to the subsequent combustion of the excess of methane in the air. Reduction of the diameter of the aperture increased the length of the projected flame for mixtures containing an excess of methane, except for very small apertures, but decreased the length for the weak mixtures. The projection of the flame into an atmosphere of carbon dioxide was shorter than in air, being about three times the length of the original column of explosive mixture with an unconstricted tube. Reduction of the diameter of the aperture decreased the distance of projection of the flame into carbon dioxide. The use of carbon dioxide at the mouth of a stopping when sealing off a gob fire is suggested in order to minimise the distance of projection of flame should an explosion occur behind the stopping. A. B. MANNING.

Detection of firedamp by electrical means. C. S. W. GRICE and A. G. GULLIFORD (Fuel, 1926, **5**, 364—368).—To indicate the presence of methane to the user of a miner's electric lamp, a piece of platinum fuse wire 3 mm. long and 0·35 mm. diameter, is fitted into the bulb circuit of the lamp, so that, when an electric current of 2·0 volts is passed through, it will fuse if exposed to an atmosphere containing 3% of methane. The fuse wire is held in a replaceable fuse-holder which fits into two socket contacts in the base of a perforated brass cylinder, protected by wire gauzes, attached to the side of the lamp. A switch is also provided to relight the bulb after the circuit is broken. The detector is inaccurate for voltages below 1·8, but is reliable for the normal 8-hour discharge of a 2-volt accumulator in good condition. A. C. MONKHOUSE.

Removal of carbon dioxide from industrial gases by washing [with water] at ordinary pressure. F. FISCHER and P. DILTHEY (Brennstoff-Chem., 1926, **7**, 277—282).—Water is fed in at the top of a tower, packed with coke or Raschig rings, up which the gases pass, and, having taken up carbon dioxide, flows by gravity from the bottom of the tower into the top of another, exactly similar, tower up which air is passed, and in which the carbon dioxide is again expelled from the water, the latter being returned, in closed circuit, to the first tower. For optimum results, with a gas mixture containing about 20% of carbon dioxide and at a temperature of about 20°, the ratio of gas : air : water is 1 : 10 : 2 by volume, and an effective tower space of 0·5 cub. m. is required to wash 1 cub. m. of gas per hour. Under these conditions an initial gas mixture containing 21·9% of carbon dioxide, 75·9% of carbon monoxide and hydrogen (together), 0·2% of oxygen, and 2·0% of nitrogen yields a washed gas containing 2·6% of carbon dioxide, 89·7% of carbon monoxide and hydrogen (together), 1·6% of oxygen, and 6·1% of nitrogen. Coke is a rather more efficient tower packing than Raschig rings. W. T. K. BRAUNHOLTZ.

Determination of free sulphur in spent gas-purifying material. D. STAVORINUS (Gas- u. Wasserf., 1926, **69**, 790—791).—Methods of determining sulphur in spent oxide are criticised. The removal of tar from the sulphur when extracted by carbon disulphide has

been effected at the Rotterdam gasworks by adsorption on active charcoals, the most suitable being finely powdered "medicinal norit." A piece of glass tubing drawn to a fine point is packed with a piece of cotton wool pressed gently down; on to this is placed a charge of active carbon, preferably "medicinal norit," and then a weighed sample (10 g.) of the spent oxide under test. The tube is clamped upright with its end dipping into a tared evaporating basin. Carbon disulphide is poured in to fill the tube; the tarry and oily matters are retained on the active carbon, and the sulphur solution passes into the dish, where the solvent evaporates. Further lots of carbon disulphide are poured through the tube until in all 150 c.c. have been used.

R. A. A. Taylor.

Cracking of tars and mineral oils at atmospheric pressure without addition of hydrogen, compared with the Bergius process. H. Herbst (Petroleum, 1926, 25, 947—949; cf. László, B., 1926, 523).—In the "berginisation" of lignite mixed with coal tar, the tar cannot be recovered as such. It may be advantageous to crack the coal tar and the tar obtained from lignite, at ordinary pressures without hydrogen. The coke can be gasified and worked up into synthetic petroleum-like products by known methods. The high temperature of berginisation favours the elimination of hydrogen. The unsaturated compounds arising from the original cracking process may be converted into saturated substances by treatment with hydrogen at a comparatively low pressure. Removal of unsaturated compounds by the Edeleanu process has the disadvantage that traces of sulphur dioxide are difficult to remove, and extraction with organic solvents is preferable. W. N. Hoyte.

See also A., Oct., 1018, **Detection and determination of carbon monoxide** (Kast and Selle).

Refractories for gas plants. Cole.—See VIII.

Patents.

Manufacture of agglomerated fuel. L. Liais (E.P. 251,605, 9.4.26. Conv., 30.4.25).—Sodium bicarbonate, either solid or in solution, is added to the pitch or tar binder used in making briquettes. The effervescence produced by the carbon dioxide generated in the binder causes it to spread more efficiently over the particles of the briquetted material, and the proportion of binder required to produce the same strength is consequently reduced. R. A. A. Taylor.

Production of finely divided carbon at a low temperature. Braunkohlen Produkte A.-G. (G.P. 431,933, 29.5.24).—Producer tar or crude oils rich in asphalt, if necessary with the addition of charcoal or wood, are heated to 300—500° under pressure so that a part of the oil remains liquid during the reaction. The resulting carbon may be purified by washing with hot water. Alternatively the oily raw material is heated to 300—500°, and is passed through a tube which is partly throttled. W. G. Carey.

Method of burning pulverised fuel. A. L. Mond. From E. H. Peabody (E.P. 256,685, 12.5.25).—The main body of air, supplied either by forced or induced draught, is put in rotation by a series of vanes and enters the furnace through a bell-mouth formed in the furnace wall. Just behind the furnace wall the pulverised fuel suspended in air enters in the form of a thin sheet of annular form, which also rotates and enters the main stream of air from the outside at an angle to the axis of flow. Just behind the pulverised fuel entrance is a central oil burner for starting or maintaining combustion. B. M. Venables.

Operating coke ovens. R. B. Kernohan (U.S.P. 1,596,048, 17.8.26. Appl., 12.9.24).—A horizontal coke oven is divided into two vertical chambers by a non-combustible core. Heating takes place both from the core and from the oven walls, causing vertical planes of cleavage in the coke in both chambers.

A. C. Monkhouse.

Coke oven. M. W. Ditto, Assr. to Chicago Trust Co. (U.S.P. 1,596,408—9, 17.8.26. Appl., 14.1.22. Renewed 8.1.26).—(A) The oven is constructed with a series of U-shaped flues, with vertical arms in the two oven walls and a cross connexion in the sole structure. The combustion chambers are at the upper end of each arm, with connexions to the regenerators for the admission of air and gas and the removal of waste gas. (B) In the heating walls of the oven is arranged a mesh of interconnected flues which transfer the gases horizontally to a series of combustion chambers at each end of the wall in which are gas nozzles. A series of laterally spaced ports connect the heating flues between the combustion chambers.

A. C. Monkhouse.

Apparatus for extracting gas from bituminous materials. E. W. Hartman, Assr. to Hartman Interests, Inc. (U.S.P. 1,595,301, 10.8.26. Appl., 31.5.21).—An apparatus for gasifying solid or liquid bituminous material, such as shale or heavy petroleum, consists of a cylindrical retort with a vertical axis fixed on an annular firebox arranged to heat all the bottom plate. Mounted in the axis of the retort is a hollow vertical rotating shaft, which drives a series of rakes adapted to stir the charge on the bottom of the retort, and through which the gas formed is removed. Means are provided for feeding the solid or liquid material into the retort, and for withdrawing residue when required. T. S. Wheeler.

Continuous carbonisation and cracking of bituminous materials under pressure. K. Bube (E.P. 256,845, 22.12.25).—A pulp obtained by mixing bituminous materials, *e.g.*, lignite, with liquid hydrocarbons, *e.g.*, lignite producer tar, is pumped through vessels capable of resisting pressure at or above 300° The process is continuous, the products being withdrawn so that the level in the reaction vessel is kept constant.

A. C. Monkhouse.

Gas generators. F. Umpleby (E.P. 257,023, 15.8.25).—The plant consists of a central cylindrical chamber, in which any suitable solid or liquid fuel may be gasified by means of air or steam, or both. The chamber is surrounded for heating purposes by surface-combustion burners made of H-shaped bricks of refractory material, arranged polygonally, which form, with the wall of the generating chamber, a channel for air, and, with the outer casing, a channel for fuel gas.

The products of combustion pass down a channel in the wall of the generating chamber, and communicate their heat to it, and they may pass into the generator.

R. A. A. Taylor.

Production of hydrogen from water-gas and steam. M. Fränkl (G.P. 431,758, 7.7.25).—A temperature of 1500—1800° is maintained continuously in a highly refractory furnace by using pure oxygen with a powdered coal, or a gas, flame. A mixture of water-gas and steam is passed through the furnace, the carbon monoxide reacting with the steam to produce hydrogen and carbon dioxide. W. G. Carey.

Thermal decomposition of gaseous hydrocarbons, natural gas, etc. by heat. I. Szarvasy (U.S.P. 1,592,474, 13.7.26. Appl., 23.2.24).—Gaseous hydrocarbons are passed into a chamber maintained at decomposing temperature, and provided with pervious walls. The chamber may be heated by gases passing through the walls and burning in proximity to them.

H. Moore.

Petroleum still. L. J. Husted, Assr. to F. L. Rounsevell (U.S.P. 1,596,660, 17.8.26. Appl., 4.3.20. Renewed 28.12.25).—A series of horizontal cylindrical stills are connected end to end, division walls being left between the adjacent stills. A group of pipes runs parallel to the axis of the stills from end to end of the series, and hot liquid is forced through the pipes. In each still petroleum is sprayed upwards against the hot pipes, the vapours are condensed separately, and the residue is drawn off. W. N. Hoyte.

Cracking petroleum oil. C. P. Dubbs, Assr. to Universal Oil Products Co. (U.S.P. 1,592,560, 13.7.26. Appl. 27.11.22).—The oil is passed in a restricted stream through the coolest part of a heating zone, and then united with the reflux condensate formed in the process. The mixture is passed in a restricted stream through the hottest part of the heating zone, and thence to an enlarged reaction zone, from which no oil returns to the stream mentioned. The vapours from the enlarged zone are passed to a dephlegmator, where part is condensed to form the reflux condensate, while the vapours pass to a condenser. Pressure above atmospheric is maintained on the oil. H. Moore.

Cracking hydrocarbons. E. C. Herthel and E. W. Isom, Assrs. to Sinclair Refining Co. (U.S.P. 1,592,489, 13.7.26. Appl., 20.11.24).—Oil is circulated to and from a body of oil in a supply tank through a heater in which it is raised to cracking temperature. The oil from the heater is passed to the upper stratum of the tank, while oil is withdrawn from the lower stratum for recirculation through the heater. Free flow downwards in the tank is obstructed, causing stratification of the oil.

H. Moore.

Cracking of hydrocarbons. Sinclair Refining Co., Assees. of E. C. Herthel and H. L. Pelzer (E.P. 246,116, 22.12.24. Conv., 17.1.25; cf. B., 1925, 907; 1926, 352). —In pressure tube or shell stills for cracking heavier oils to produce motor spirit, oxides, such as copper oxide, but not iron oxide or bauxite, are introduced into the oil, which is continuously circulated through them. The oxides are supported on a bed of asbestos, firebrick, coke, fuller's earth, etc. on a perforated screen. They exercise a selective action, retaining asphaltic and sulphur-containing substances which tend to form deposits on the surfaces of the still. The oil containing an excess of asphaltic substances may be gradually withdrawn and replaced by fresh charging stock. H. Moore.

Distillation of lubricating oils from mineral oils. Simplex Refining Co., Assees. of G. A. Kramer (E.P. 251,928, 5.8.25. Conv., 9.5.25).—The oil to be distilled, preferably one topped to 320°, is preheated and charged into a still, in which pressure is reduced to and maintained at about 5 mm.; external heat is applied and steam is blown in so that the vapours contain about 10% by weight of steam. An air condenser causes the temperature of the vapours to drop 6—30°, and so to deposit the lubricating oil, which is drawn off. Some steam and oil of low b.p. are then condensed in a water condenser, and a steam ejector raises the pressure of the vapours to about 50 mm., when the remainder of the steam and oil are condensed. W. N. Hoyte.

Process for breaking water-in-oil [petroleum] emulsions. M. De Groote, Assr. to W. S. Barnickel & Co. (U.S.P. 1,596,585—1,596,598, 17.8.26. Appl., 23.12.24, 6.4.25, 29.5.25, 1 and 7.10.25).—The following means are employed in breaking emulsions: passing the emulsion through a non-absorbent material, such as pure zinc, on which a demulsifying agent is held by adsorption; filtering the emulsion mixed with demulsifying agent through a bed of sand; treating emulsions with equal quantities of non-saponaceous demulsifying agent and a substance capable of forming brine-in-oil emulsions; treating emulsions with a soap obtained from a shale oil sludge or a sulphonated shale oil sludge; treatment with a lactone or a sulphonated condensation product containing a polycyclic aromatic compound and a fatty material, or with an ester of a naphthenic acid or with the condensation product of a sulphonated naphthenic acid and an aromatic hydrocarbon; treatment with an oil-soluble hydrophobe compound in which water has been combined by a hydrophobe soap; bringing the emulsion to incipient breaking point without dilution by treating with a suitable agent and then added further emulsion; treating emulsion with a highly colloidal coagulescent suspension containing a soap-forming radical. W. N. Hoyte.

Apparatus for recovering heat from coke, slags, ashes, and other solid substances. P. Bringhenti (U.S.P. 1,597,718, 31.8.26. Appl., 9.6.24. Conv., 18.7.23).—See E.P. 228,406; B., 1925, 232.

Complete gasification of bituminous fuel. A. Breisig (U.S.P. 1,598,473, 31.8.26. Appl., 25.9.22. Conv., 25.5.21).—See E.P. 207,651; B., 1924, 122.

Purification of gas. Humphreys & Glasgow, Ltd., Assees. of W. H. Fulweiler (E.P. 248,355, 15.2.26. Conv., 27.2.25).—See U.S.P. 1,589,749; B., 1926, 699.

Removing tar from the vapours arising from the carbonization of wood. E. A. Barbet (U.S.P. 1,598,547, 31.8.26. Appl., 20.10.21. Conv., 21.10.20). —See E.P. 170,591; B., 1923, 343 A.

Removing hydrogen sulphide from gases

W. GLUUD (U.S.P. 1,597,964, 31.8.26. Appl., 11.9.22. Conv., 20.9.21).—See E.P. 186,316; B., 1923, 887 A.

Liquid fuel burners. W. A. HEDWORTH (E.P. 257,687, 11.6.25).

Atmospheric gas and oil burners. C. M. HUNTER (E.P. 257,416, 18.9.25).

Carbon black, gases, etc. (U.S.P. 1,597,277).—See XIII.

III.—TAR AND TAR PRODUCTS.

Cracking of tars. HERBST.—See II.

PATENTS.

Tar distillation apparatus. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,596,050, 17.8.26. Appl., 6.7.21).—A tar boiler is fitted with longitudinal fire tubes joined by removable sections outside the boiler. The tubes are connected at one end to the source of gas supply and at the other to the flue. Provision is made for the expansion of the boiler during distillation. A. C. MONKHOUSE.

Decreosotising tars and tar products. K. BUBE (E.P. 257,151, 22.12.25).—The material is extracted with mixtures of alcohol and water containing more water than alcohol, at temperatures above 50°, under the ordinary or slightly raised pressure. Alcohol is recovered from the solution by distillation without the use of a dephl·gmator, until the creosote constituents separate from the residual liquor. For example, lignite tar oil containing 14% of creosote is extracted at 83° with a mixture of 75 pts. of water and 25 pts. of alcohol; the residual oil then contains 3% of creosote. L. A. COLES.

Manufacture of prepared tar for road-making, etc. L. H. SENSICLE (E.P. 257,024, 21.5.25).—Tar is charged at 55—60° into a high-speed centrifugal separator, after which the moisture content is further reduced to 0·5% or less by passing a current of cold air through the tar maintained at a temperature sufficiently high to remove moisture but not to cause chemical action, *e.g.*, at about 60°, or at a somewhat higher temperature if it is desired to remove naphthalene and light oils as well as water. Bitumen and solid material removed from the bowl of the centrifuge, as well as other materials, such as tar distillates or pitch, may be added to the tar during the air treatment. L. A. COLES.

IV.—DYESTUFFS AND INTERMEDIATES.

Action of chlorine on aniline. Formation of induline dyestuffs. I. D. G. BROWN and W. M. CUMMING (J.S.C.I., 1926, **45**, 355—360 T).—The action of chlorine on aqueous aniline (3%) at ordinary temperature results in the formation of azophenine contaminated with aniline-black. At low temperature in an inert water-free solvent, such as carbon tetrachloride, an almost theoretical yield of *s*-trichloroaniline is obtained. The action of chlorine on boiling aniline produces ultimately an induline dyestuff with evolution of hydrogen chloride. This induline, which is the hydrochloride of a base, can be recrystallised from alcohol, the free base being liberated by means of alkalis. The induline in acetic acid solution gives a yellow leuco-compound with sodium hyposulphite (hydrosulphite), which is almost immediately reoxidised in air. When the induline is heated with fuming hydrochloric acid at 300° in a sealed tube, aniline and diphenylamine (the latter by a side reaction) are formed as well as a greenish-black substance with characteristic fluorescence. The parent substance of the dyestuff is therefore very stable. From an investigation of the high-temperature chlorination it was suspected that the chloro-substituted anilines were responsible for the formation of the dyestuff. It was found ultimately that *p*-chloroaniline when heated with aniline hydrochloride to melting point, forms an induline with evolution of ammonia. The analytical figures indicate that the formula for the hydrochloride prepared by direct chlorination is $C_{36}H_{26}N_6Cl_2,2HCl$, whilst the base from *p*-chloroaniline is $C_{36}H_{27}N_6Cl$. It is therefore believed that the parent substance contains a phenazine nucleus, and is represented by $C_{36}H_{28}N_6$, the nucleus being $C_{12}H_4N_2$. Suggested configurations for the molecule are given and discussed. Absorption spectra of the two dyestuffs indicate that the configuration is the same in each case. The dyeing properties of the hydrochloride and base are described.

Identification of naphthalenoid reduction products of azo dyes. R. B. FORSTER and T. H. HANSON (J. Soc. Dyers and Col., 1926, **42**, 272—275).—Reduction is effected by acid stannous chloride or hyposulphite. The ammoniacal solutions of the azo coupling components, chiefly aminonaphtholsulphonic acids, are spotted on filter paper with hydrochloric acid, ferric chloride, vanadium chloride, potassium dichromate, nickel chloride, uranium sulphate, potassium ferrocyanide, potassium ferricyanide, copper sulphate, Schweizer's reagent, ferrous ammonium sulphate, cobalt nitrate, and silver nitrate. Many of these give well-defined colour reactions, which are tabulated, and described in detail. J. S. H. DAVIES.

Method for distinguishing α- and β-naphthols. F. LEWISCH (Textilber., 1926, **7**, 863).—A carmine-red coloration develops immediately when 10 c.c. of concentrated sulphuric acid are poured over 0·1 g. of α-naphthol in the presence of a piece of bleached cotton, the coloration changing to a permanent deep reddish-violet within 1 hr. Under similar conditions, β-naphthol yields a brown coloration which changes to bluish-green. When the coloured solutions are diluted six times, the reddish-violet and bluish-green colorations change to bright violet and green respectively. These reactions are used to detect the presence of traces of α-naphthol in β-naphthol or *vice versa*. A. J. HALL.

See also A., Oct., 1028, **2:3:4-Trinitrotoluene** (GORNALL and ROBINSON). 1030, **o-Toluidine** (TANNER and LASSELLE). 1031, **Optically active dyes** (BRODE and ADAMS). 1032, **Derivatives of benzene- and toluene-azonaphthylamine** (TRÖGER and SCHAEFER). 1043, **Delphinidin chloride 3-methyl ether** (GATEWOOD and ROBINSON); **Synthesis of peonidin chloride** (NOLAN and others). 1049, **Determination of methoxyl** (NIERENSTEIN); **Determination of primary aromatic amines** (GRIGORJEV). 1050, **Determination of benzidine and its analogues** (HERZOG).

PATENTS.

Manufacture of [chromed azo] dyestuffs. SOC. CHEM. IND. IN BASLE (E.P. 247,556, 9.2.26. Conv., 14.2.25).—Azo dyes derived by coupling a nitrated *o*-hydroxydiazo compound with an α-naphthylamine substituted in the *ortho*-position to the amino-group are treated with salts, oxides, or hydroxides of tervalent chromium. For example, 48·9 pts. of the sodium salt of the azo dye obtained from picramic acid and naphthionic acid are dissolved in 900 parts of boiling water, 18 pts. of chromium oxide in the form of a 4% solution of chromium fluoride are added, the whole is boiled under reflux in presence of glass powder for 18—24 hrs., and the new *dye* salted out. It dyes wool a fast dark olive-green shade. A. DAVIDSON.

Manufacture of [azo] dyestuffs containing chromium. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (E.P. 257,820, 4.2.26).—A solution of the azo dye from 6-nitro-2-aminophenol-4-sulphonic acid and β-naphthol is boiled under reflux with a solution of chromium fluoride in presence of glass powder. The *dye*, which contains chromium, is separated by evaporation and salting out. Similar dyes are obtained by using other agents yielding chromium such as chromium acetate or formate or neutral freshly precipitated chromium hydroxide. The products dye wool in reddish-black shades fast to light and fulling. (Cf. E.P. 26,460 of 1912 and 116,535, B., 1913, 905; 1918, 460 A.) A. DAVIDSON.

Manufacture of [azo] dyestuffs [from barbituric acids]. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (E.P. 257,797, 15.12.25).—Barbituric acids of the general formula $R(N{<}{}^{CO\cdot NH}_{CO\cdot CH_2}{>}CO)_n$, in which R = H, alkyl, aryl, or alkaryl, and $n = 1$ or 2, are coupled with an *o*-hydroxydiazo compound. The products are acid mordant dyes which dye wool in red to brown shades which change, when chromed, to brown and red shades fast to fulling and potting and very fast to light. The dyes themselves, by treatment with chroming agents such as oxides, hydroxides, or salts of tervalent chromium, yield acid dyes which dye wool in uniform fast shades of yellow to red and rose. For example, 233 pts. of 4-sulpho-2-aminophenol-6-carboxylic acid are diazotised and added to a mixture of 134 pts. of barbituric acid, 133 pts. of 30% sodium hydroxide solution, 170 pts. of sodium carbonate, and 1500 pts. of water. After stirring for several hours, the *dye* is filtered off. The preparation of a chromed dye is carried out, for example, by boiling a solution of 37·2 pts. of the above dye in 600 pts. of water with a solution of chromium formate corresponding with 15·2 pts. of Cr_2O_3. The barbituric acids are obtained by condensing carbamides of the general formula $R(NH\cdot CO\cdot NH_2)_n$ in which R and n have the significance already mentioned, with a malonic ester. A. DAVIDSON.

Yellowish-red azo dyestuffs. H. WAGNER and A. FUNKE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,595,269, 10.8.26. Appl., 8.10.25).—Diazotised *m*-nitro-*p*-aminophenyl alkyl ethers, especially the ethyl or methyl derivatives, are coupled with an *o*-alkyl- or a *p*-alkoxy-anilide of acetoacetic acid, *e.g.*, acetoacet-*o*-toluidide, -*p*-anisidide, or -*p*-phenetidide, to give products which dye fabrics yellowish-red shades, which in the form of lakes are very fast to light. T. S. WHEELER.

Manufacture of anthraquinone dyestuffs possessing affinity for acetyl silk. BRIT. DYESTUFFS CORP., LTD., W. H. PERKIN, and C. HOLLINS (E.P. 257,353, 4.6.25).—By the condensation of a primary anthraquinonylamine with α-aminoanthraquinone, or α-diaminoanthraquinones, or simple derivatives of these such as diaminoanthrarufin or diaminochrysazin, in presence of anhydrous calcium chloride or iodine as condensing agent, at 200—300°, new *substances* are obtained which are probably anthraquinonyliminoanthrones. The products dye acetyl silk from an aqueous suspension. A. DAVIDSON.

Anthracene dye [from dihalogenopyranthrones]. I. GUBELMANN, Assr. to NEWPORT CO. (U.S.P. 1,596,528—31, 17.8.26. Appl., [A, B] 4.4.25, [C, D] 9.5.25).—Dichloro- or dibromo-pyranthrone is heated with (A) 1-aminoanthraquinone or a derivative such as 1-amino-2-methylanthraquinone or 6-chloro-1-aminoanthraquinone, (B) 2-aminoanthraquinone or a derivative, (C) carbazole, or (D) aminodibenzanthrone, when condensation occurs, one of the halogen atoms of the pyranthrone derivative being eliminated with a hydrogen atom attached to the nitrogen atom in the other compound. The products dye cotton from a hyposulphite vat violet (A, B, C) or dull bluish-violet (D) shades oxidising in air or on treatment with oxidising agents to rich brown shades fast to washing and light. Reaction is effected by heating the components at about 200° for about 15 hrs. in a suitable liquid such as nitrobenzene or naphthalene in presence of an acid-binding agent, such as anhydrous sodium carbonate or acetate, and of a catalyst such as basic copper acetate. In the reactions described under (C) and (D) the shades obtained vary slightly with the time of heating. T. S. WHEELER.

Manufacture of phenols [from benzene hydrocarbons]. W. J. HALE, Assr. to DOW CHEMICAL CO. (U.S.P. 1,595,299, 10.8.26. Appl., 7.1.24).—An autoclave two-thirds filled with an emulsion of equal volumes of 20% aqueous caustic soda solution and benzene containing an oxide of uranium, with or without small proportions of oxides of vanadium, molybdenum, and tungsten, is heated at 320—400° for 1 hr., the internal pressure being maintained at about 200 atm. The oxygen in the air above the liquid in the autoclave is absorbed with formation of phenol, which dissolves in the alkali. Only a small proportion of the benzene is converted in each operation, but as there are no side reactions, that remaining after separation from the alkaline liquid can at once be used again. T. S. WHEELER.

Azo colouring matter dyeing wool fast to milling. J. BADDILEY, J. HILL, and A. RILEY, Assrs. to BRITISH DYESTUFFS CORP., LTD. (U.S.P. 1,598,109, 31.8.26. Appl., 21.9.25. Conv., 17.9.24).—See E.P. 243,115; B., 1926, 85.

Azo dyestuffs and intermediates. E. B. HIGGINS (U.S.P. 1,597,791, 31.8.26. Appl., 29.11.24. Conv., 19.12.23).—See E.P. 230,920; B., 1925, 437.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Distribution of nitrogen in the flax plant and its elimination from flax during manufacture. J. W. PORTER (J.S.C.I., 1926, **45**, 335—337 T).—The nitrogen content of flax at all stages of manufacture from the dried flax straw to the bleached yarn is given. The nitrogen is located in the cortical and epidermal cells, and from the nitrogen figures deductions are made as to the completeness of the bleaching operations. Reference is also made to the potash and phosphate content of the cortical and epidermal cells.

Pectin contents of flax. W. KIND (Spär- u. Wirtsch., 1926, **17**, 12; Textilber., 1926, **7**, 872).—Irish water-retted, Courtrai, Russian, and Peufaillit-retted flax contained 7·02, 4·46, 6·45, and 5·78% of pectins respectively. A brown powder containing 84·67% of pectin was obtained by extracting flax stalks with a 1% solution of ammonium oxalate. Pectins remaining in bleached linen have a considerable influence on its lustre, appearance, and tensile strength. A. J. HALL.

Load-elongation curves and lustre of artificial silk. K. ATSUKI (Cellulose Ind., Tokyo, 1926, **2**, 218—226).—The load-elongation curves have been determined for three types of Japanese viscose silks, wet and dry, as compared with cotton and natural silk. For the artificial silks the tensile strength is lower, the elongation is greater, and the loss of strength on wetting is greater than for cotton. The ratio stress : strain is much lower for viscose silk than for cotton. These inferior mechanical properties are attributed to the modification of the cellulose, the heterogeneity of the cellulose particles and the irregularity of their arrangement, and to the presence of lower grade impurities such as β-cellulose. The load-elongation curves of the different samples of viscose silk vary considerably, especially under small loads, some showing a pronounced elastic limit and yield point, whilst one had a smooth curve right up to the break point without definite elastic limit or yield point. The latter had a soft damp feel considered to be inferior for weaving purposes. The form of the curve may be considerably influenced by the ageing and conditions of coagulation of the viscose. The lustre is influenced by the surface structure, the fineness of the filament, and the transparency or the arrangement and size of the cellulose particles and the existence of minute gas cavities. J. F. BRIGGS.

Valuation of artificial silks. N. U. BERCHIN (Chem.-Ztg., 1926, **50**, 643).—The numbers obtained by multiplying together the elasticity (expressed as a percentage) and the wet and dry tensile strengths (expressed in g. per denier) of artificial or natural silk yarns are considered to be suitable for use in comparing their commercial value. For example, artificial silk yarn having 20% elasticity, 2·0 and 0·65 g. tensile strength dry and wet respectively ($20 \times 2{\cdot}0 \times 0{\cdot}65 = 26$), is considered superior to artificial silk yarn having 25% elasticity, 1·3 and 0·55 g. tensile strength dry and wet respectively ($25 \times 1{\cdot}3 \times 0{\cdot}55 = 17{\cdot}67$). The method of valuation may be extended so as to include lustre and purity of white colour. A. J. HALL.

Action of oxygen on alkali-cellulose. W. WELTZIEN and G. ZUM TOBEL (Papier-Fabr., 1926, **24**, 413—414).—Cotton or artificial silk cellulose, when swollen with sodium hydroxide, absorbs oxygen freely, even at the ordinary temperature, and the reaction becomes very pronounced at 40°. Observations made with cotton impregnated with 3 times its weight of 10% sodium hydroxide showed a steady absorption of oxygen at 60°, amounting to 2 atoms of oxygen per mol. of $C_6H_{10}O_5$ after 35 days, with no indication of any break in the process after further exposure. J. F. BRIGGS.

Chemistry of the sulphite wood pulp digestion. E. HÄGGLUND (Papier-Fabr., 1926, **24**, 449—450, 483—488; cf. B., 1925, 238, 586; 1926, 312).—In the first stage of the digestion there is an absorption of the bisulphite from the liquor to form an insoluble lignosulphonic acid or salt in the wood, which is later liberated by acid hydrolysis from its union with the carbohydrates and enters the liquid phase in the soluble form. The lignin of under-digested sulphite pulp exists in the form of the insoluble lignosulphonic acid; it possesses fluorescent properties; it cannot be extracted by washing with dilute alkali, but on boiling with 1% hydrochloric acid it is hydrolysed, rapidly at first, but later very slowly. After boiling with acid for 1 hr., 65% of the contained sulphur and methoxyl of the crude sulphite pulp had been removed, but after 8 hrs. the residue still retained 18% of the original lignosulphonic acid contents. Hence it appears that simultaneously with hydrolysis, under the prolonged action of acids, the lignosulphonic acid undergoes irreversible polymerisation. To this polymerisation, with its attendant resinification, the phenomenon of "blackened," over-digested charges may be attributed. The hydrogen-ion concentration of the bisulphite liquor may be measured by the sucrose-inversion method; it decreases in the early stage while bisulphite is being fixed by the lignin of the wood, and increases later during the hydrolysis stage. The pentosans are hydrolysed at first more rapidly than the hexosans, but later the latter are more profoundly attacked. Comparative digestions with calcium, magnesium, and ammonium bisulphites showed but slight differences in chemical mechanism.

J. F. BRIGGS.

Violet fluorescence of sulphite wood pulp and waste liquors. O. GERNGROSS and K. TSOU (Papier-Fabr., 1926, **24**, 497—499).—Sulphite wood liquors and unbleached sulphite pulp show a strong lilac fluorescence in ultra-violet light, which changes to bright green by the action of alkali, and is gradually destroyed during bleaching. The substance with this property has been located in the free state in the living secondary bark layers of fresh spruce wood, but it also exists in a latent and firmly combined form in the wood itself. The fluorescence is observed in the light of a Hanau analysis quartz lamp and shows up very strongly when the outer bark is lifted with a knife. Aqueous extracts of spruce bark, as used for tanning, are exceptionally rich in this fluorescent substance and show the phenomenon even when highly diluted. From acid or neutral solutions the fluorescent substance is irreversibly absorbed by cellulose fibres, both in spruce-bark extracts and in sulphite waste liquors, although in the latter the fixation properties on

cellulose are considerably weakened. Although the free, water-soluble fluorescent substance is derived only from the secondary bark layers, wood which has been carefully separated from these layers and shows none of the characteristic lilac fluorescence develops this substance when digested with calcium bisulphite at 115°. The digestion liquor acquires the same properties as ordinary sulphite waste liquor and the fibre residue is firmly "dyed" with the violet fluorescence. Simple boiling of wood with water is not sufficient to liberate the fluorescent substance, but if a piece of cotton cellulose be digested with wood and water for 8 hrs. at 110°, the cotton acquires a slight and somewhat modified violet fluorescence, which changes to bluish-green with alkali, instead of to the characteristic bright green. At the same time the wood shows no fluorescence, but the water shows the characteristics of the normal fluorescence. This proves that the fluorescence of unbleached sulphite pulp is not due to a lignosulphonic acid, but to a natural constituent of the wood. J. F. Briggs.

Testing the strength of [paper] half-stuffs. H. Schwalbe (Papier-Fabr., 1926, **24**, 465—468, 481—483).—The testing of half-stuffs is carried out by the preparation of sheets of paper under carefully standardised conditions. For routine tests in the pulp mill the fibre may be conveniently prepared in the Lampen ball mill, but for the complete investigation of a new type of pulp the ball mill must be supplemented by beating trials in a properly designed hollander capable of treating stuff at the normal 5% concentration. The hollander tests must be controlled by determinations of the degree of beating by sedimentation or Schopper's apparatus. The formation of the sheets is the most difficult operation, and accurate conditions must be established. Sheets should be made at a substance of 100 g./m.^{2} The pressure used for compacting the sheets has a great influence on the strength of the paper; a press adjusted at 35 kg./cm.^{2} is used. Conditions of drying the sheets, preferably on a heated metal surface, must be standardised; careful adjustment of atmospheric humidity is necessary when performing the tensile tests.
J. F. Briggs.

Aluminium resinate in the sizing of paper. E. Oman (Papier-Fabr., 1926, **24**, 410—413, 451—455).—Aluminium resinate, freshly prepared and dried without heat, is completely soluble in ether, benzene, and carbon tetrachloride; it is insoluble in alcohol, which decomposes it, dissolving only a portion of free resin acid. It also undergoes decomposition on exposure to heat, after which ether selectively extracts a portion of free resin acid and the aluminium resinate becomes insoluble. Aluminium resinate is very sensitive to acid conditions; a hydrogen-ion concentration of p_H 3·5 equivalent to the degree of acidity in the beater liberates a considerable proportion of free resin acid. Sulphite pulp also modifies the precipitated aluminium resinate so that it is no longer completely soluble in ether even when dried at the ordinary temperature, whilst the pulp after drying at 115° only yields 40—55% of its total resin on extraction with ether. Acidification is necessary for the quantitative extraction of total resin. An ethereal solution of aluminium resinate is capable of sizing paper; a suspension of precipitated neutral aluminium resinate also possesses sizing properties, but gives results inferior to those obtained when a certain amount of free resin is liberated by acidification either with alum or with sulphuric acid. A hydrogen-ion concentration of p_H 5·5 produced with excess of alum gave a slightly better sizing than one of 3·5 produced with sulphuric acid. The inferiority of the neutral resinate as a sizing material is ascribed partly to the coarser dimensions of its particles and partly to the absence of sintering on the drying cylinders. Under ordinary conditions of sizing with slight acidity no important part can be ascribed to the precipitation of aluminium hydroxide in the fibre. If aluminium is fixed at all in acid conditions it is in exchange for calcium ions, and it may thus confer on the fibre a positive charge whereby the negatively charged particles of free resin are attracted.
J. F. Briggs.

See also A., Oct., 1026, **Molecular dimensions of cellulose** (Brunswig); **Constitutional formula for cellulose** (Gray); **Esterification of alkali-cellulose** (Kita and others).

Patents.

Degreasing textiles [wool]. N.V. Algem. Chem. Produktenhandel, and W. A. Meyer (E.P. 257,192, 6.4.26).—Wool is continuously degreased by treatment with organic solvents so that it retains its original strength, softness, and elasticity while felting is avoided. The greasy wool, heaped on a perforated conveyor belt, is drawn through a closed chamber and between squeezing rollers, being simultaneously sprayed with a suitable solvent such as benzene. It then passes through a drying chamber maintained under reduced pressure and the solvent vapour is withdrawn, cooled to below 0°, and the solvent recovered by passage over a suitable adsorbent. A. J. Hall.

Washing or cleaning wool, silk, soiled fabrics, and the like fibrous textile materials. E. C. Duhamel, and Comp. Gén. des Ind. Textiles (E.P. 234,055, 2.3.25. Conv., 13.5.24. Addn. to 221,521, B., 1925, 985).—In washing wool or the like with aqueous solutions or suspensions of detergents, purification of the solution by continuous centrifuging may be supplemented or replaced by other continuous treatments, *e.g.*, frothing, filtration, or electrical treatment.
A. Geake.

Manufacture of artificial silk. M. P. Lanfry and J. E. Brandenberger (E.P. 255,527, 22.4.25 and 16.2.26).—Glossy filaments with a hollow core are spun from viscose containing alkali carbonate and of any degree of ripening, the ratio of alkalinity (as NaOH) to cellulose before adding the alkali carbonate being less than 6 : 7·3. The viscose is squirted into a coagulating bath containing sulphuric acid and sodium sulphate in proportions depending on the degree of ripeness of the viscose and on the alkalinity. Example: Viscose containing 7·3% of cellulose and 4% of total alkalinity (as NaOH) is mixed with sodium carbonate to bring the alkalinity to 7%. The viscose is ripened at 13° for 73 hrs. from the beginning of the ripening stage, and is spun into a bath containing 145 g. of sulphuric acid and 228 g. of sodium sulphate per litre, at a speed of 45 m.

per min., filament denier 6, temperature 45°, length of immersion 35 cm. With a time of ripening of 110 hrs. at 13°, the concentration of the coagulating bath for the same viscose would be 125 g. of sulphuric acid and 40 g. of sodium sulphate per litre. J. F. Briggs.

Artificial silk, thread, films, ribbons, etc. from viscose. O. Faust and A. Kämpf (G.P. 431,846. 16.11.22).—Sulphonic acids of condensation products of phenolsulphonic acids with aldehydes, aldoses, ketoses, complex carbohydrates, or wood are added to viscose, or the free acids or their salts are added partly to the viscose and partly to the spinning bath. Unripe viscose may be used. A. Geake.

Method of moth-proofing. J. M. Ross and M. J. Ross, Assrs. to I. B. Ross (U.S.P. 1,594,632, 3.8.26. Appl., 3.7.25).—The fibres of the textile are softened and expanded by means of steam impregnated with volatilised benzaldehyde and phenol. J. F. Briggs.

Preparation of fur for shrinking and felting. J. H. Martin (U.S.P. 1,597,992, 31.8.26. Appl., 3.3.25). —Fur is treated with a solution of an alkali orthoborate. A. Geake.

Obtaining pure cellulose fibre from impure material containing incrusting substances. H. Silbermann (G.P. 432,052, 15.11.23. Addn. to 407,500, B., 1925, 203).—Saturated or superheated steam may be added to the gaseous chlorine with which the material is treated, and the vessel cooled after removing the steam. The material may be cooled at —10° to 0° before treatment. For the alkaline treatment, a dilute solution of a salt of a weak acid is used, and heated under a pressure of at least 1 atm. A. Geake.

Bleaching process [for paper pulp]. W. D. Gregor, W. M. Osborne, and A. J. Kemzura (U.S.P. 1,597,880, 31.8.26. Appl., 14.7.24).—Wet paper pulp is treated with insufficient bleaching agent at 21°, and, when the latter is exhausted, the pulp is washed and sufficient bleaching agent added to bleach to the required degree at 29°. A. Geake.

[Filler and coating for] paper. H. R. Rafsky, now H. R. Rafton (U.S.P. 1,595,416, 10.8.26. Appl., 3.4.22).—A mixture of finely-divided calcium carbonate and basic magnesium carbonate forms a cheap filling or coating material for paper, and gives a uniform opaque product with enhanced whiteness. It is obtained by slaking magnesian lime, and treating the product with carbon dioxide in presence of water until calcium hydrogen carbonate begins to be formed. Alternatively a soluble carbonate is added to a solution of a calcium and a magnesium salt. T. S. Wheeler.

Manufacture of artificial products from viscose. D. van der Want and M. P. A. Bouman, Assrs. to N.V. Nederlandsche Kunstzijdefabr. (U.S.P. 1,596,906, 24.8.26. Appl., 4.3.26. Conv., 5.3.25).—See E.P. 248,750; B., 1926, 871.

Paper pulp and paper. B. S. Summers (U.S.P. 1,597,840—1, 31.8.26. Appl., 9.7.24).—See Can. P. 246,537; B., 1926, 153.

Reclaiming used paper. G. Hammond (E.P. 257,774, 5.11.25).—See U.S.P. 1,572,478; B., 1926, 316.

Apparatus for treating artificial silk and other threads. S. Walker & Sons, Ltd., and G. E. Walker (E.P. 257,706, 7.7.25).

De-waxing, de-greasing, or de-oiling textile fabrics. D. McKellar and J. MacGregor (E.P. 257,491, 9.2.26).

Means for removing moisture from pulp, paper, straw boards, etc. E. E. Tasker (E.P. 257,449, 4.11.25).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Wetting-out agents [for textile materials]. W. Seck and H. Lachmann (Textilber., 1926, 7, 851—854).—Determination of the efficiency of wetting-out agents by methods dependent on the rate of sinking of yarn or fabric in an aqueous solution (cf. Auerbach; B., 1926, 705), or the spread of a solution along a horizontally suspended thread (cf. Herbig and Seyferth; B., 1926, 312) are considered unsatisfactory because they are subject to the disturbing influences of air bubbles occluded in the textile material and the humidity of the surrounding atmosphere respectively. The relative wetting-out powers of various proprietary substances were determined by comparison of the amounts of solution absorbed by similar pieces of fabric (5 × 6 cm.) when immersed for 10 sec. in 0·2, 0·4, and 0·6% solutions of the wetting-out agents, and then allowed to drain for 50 sec. after withdrawal. The wetting-out powers of Oranit pdr., Nekal, Avivan, Avirol KM, Diffusil N, Koloran K, Marseilles soap, Monopole soap, Turkey-red oil (50% fat), and Tetracarnite, decreased in the order named. The first three agents named are said to be alkyl derivatives of naphthalenesulphonic acids. A. J. Hall.

Influence of additions to the indigo vat. H. Pomeranz (Textilber., 1926, 7, 862—863).—The orange colour of an indigo vat containing nitrobenzene as described by Haller (B., 1926, 891) is probably due to oxidation of the indigo by the nitrobenzene whereby dehydroindigo (cf. Kalbe; B., 1909, 1192) is formed. A. J. Hall.

Use of Aktivin in volumetric analysis [of mordants etc.]. E. Jangmichl and J. Hackl (Textilber, 1926, 7, 850—851).—Crystalline Aktivin, $C_6H_4Me\cdot SO_2NClNa,3H_2O$, is a satisfactory oxidising agent for use in the volumetric analysis of tartar emetic and antimony lactates as used for the mordanting of cotton, and of sodium formaldehyde-hyposulphite. During oxidation, Aktivin is decomposed thus: $C_6H_4Me\cdot SO_2NClNa + H_2O = C_6H_4Me\cdot SO_2NH_2 + NaCl + O$. In the analysis of tartar emetic, a mixture of 25 c.c. a 1·6% solution of the sample of tartar emetic and 20 c.c. of a 2% solution of sodium bicarbonate is titrated with a 0·1N-solution of Aktivin (14·1 g. per litre) using starch–potassium iodide as an indicator. In the analysis of sodium formaldehyde-hyposulphite, a 0·4% solution of the sample is titrated directly with a 0·1N-solution of Aktivin, using starch–iodide as an indicator. The results obtained by means of Aktivin agree very closely with those obtained by the usual methods using iodine, titanous chloride, or hydrogen sulphide. Aqueous solutions of Aktivin do not deteriorate when protected from sunlight during storage; appreciable deterioration is

produced by sunlight. Commercial Aktivin is sufficiently pure for volumetric analysis after filtration of its aqueous solution, but it may be readily purified by crystallisation from its aqueous solution. A. J. Hall.

Patents.

Use of hyposulphites or sulphoxylates in the removal of dyes from textile fibres. C. S. Hollander, Assr. to Rohm & Haas Co. (U.S.P. 1,594,670, 3.8.26. Appl., 9.6.25).—Dyed goods are immersed in a mixture of a substantially insoluble formaldehyde-sulphoxylate and a readily soluble substantially neutral salt of a substance having a strongly alkaline hydroxide. J. F. Briggs.

Finishing cotton yarn. J. G. A. Rhodin, and E. B. Westman, Ltd. (E.P. 257,097, 20.8.25).—Cotton yarn under tension is impregnated or coated with a solution of cellulose (*e.g.*, cellulose acetate), then drawn through sapphire dies or between fluted rollers whereby excess of the impregnating solution is removed and the tension on the yarn is controlled, dried, and wound on bobbins. The treatment may involve simultaneous mercerisation of the cotton yarn. A. J. Hall.

Dyeing or coloration of cellulose acetate. British Celanese, Ltd., and G. H. Ellis (E.P. 255,962, 2.5.25). —Oxidation shades from fawn to full black are produced on cellulose acetate, without impairing handle or lustre, by first applying a free amino-base solubilised or dispersed in water, then preferably rinsing and impregnating the goods with a solution of suitable oxidising and catalytic agents, drying without rinsing, and "ageing" by exposure to warm moist air or steam. Alternatively, the amino-base may be applied in the form of its water-soluble salts. Example: To produce a full black, 0·75 lb. of *p*-aminodiphenylamine is intimately mixed with 6 lb. of Turkey-red oil by heating at 80° and the mixture is dispersed in 25 gals. of soft water at 80°; 10 lb. of cellulose acetate yarn are worked in the liquor as for dyeing for about 2 hrs. and then rinsed off. The yarn is then impregnated with about its own weight of a solution containing 2 pts. of gum tragacanth, 8 of chromium chloride, 4 of aluminium chloride, 3 of cupric chloride, and 60 of sodium chlorate, each separately dissolved, and made up with water to a total of 1000 pts. The impregnated goods are preferably dried at a moderate temperature and "aged" for 5 min. in a steam ager, then washed off in hot water or very dilute formic acid solution. J. F. Briggs.

Apparatus for acidulating textile and other material. E. Molinghen and J. Gaye (E.P. 245,466, 2.1.26. Conv., 2.1.25).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Preparation of sulphuryl chloride. T. H. Durrans (J.S.C.I., 1926, **45**, 347—350 T).—The rate of formation of sulphuryl chloride by the interaction of sulphur dioxide and chlorine is greatly accelerated by certain organic substances. The most potent catalysts belong to that class of esters which are compounds of saturated alcohols or of monohydric phenols and monobasic aliphatic acids containing carbon, hydrogen, and oxygen only; certain other esters, ethers, and ketones have also been found to act catalytically.

Effect of time and temperature of burning on the properties of lime. R. T. Haslam and E. C. Hermann (Ind. Eng. Chem., 1926, **18**, 960—963).—The limestones investigated were calcined in an electric resistance furnace for 2—6 hours at temperatures from 980° to 1320° and from the quicklime, hydrate was formed containing 50% excess of water. Plasticity of the hydrate when mixed with water was determined in an improved form of the Emley plasticimeter, whilst the rate of slaking was found by noting the time taken for the lime when mixed with water to reach a maximum temperature. Rate of settling of the hydrate in water, the volume of putty formed, and the rate of reaction with hydrochloric acid were also noted. The results showed that the temperature and the time of burning are important, hydrates with the maximum plasticity being produced when the limestones examined were burnt at 1090° for 4 hrs. Fineness of the hydrate particles, as shown by greater rate of reaction with acid and slower rate of settling, is also an important factor in producing plasticity. W. G. Carey.

Separation of potassium nitrate and recovery of other salts from crude Indian saltpetre. M. A. Hamid (J.S.C.I., 1926, **45**, 315—320 T).—The author discusses the application of modern phase-rule methods to the refining of crude Indian saltpetre. The requisite processes are based on a study of the isotherms at 25° and 90° of the ternary systems H_2O–K_2SO_4–KNO_3, H_2O–$NaNO_3$–KNO_3, H_2O–Na_2SO_4–$NaNO_3$, and the quaternary systems $NaNO_3$–KCl–KNO_3–$NaCl$–H_2O and Na_2SO_4–KNO_3–K_2SO_4–$NaNO_3$–H_2O.

See also A., Oct., 1000, **Partial pressures of nitric and hydrochloric acids** (Klemenc and Nagel). 1011, **Evolution of hydrochloric acid from chloride solutions** (Bodnár and Roth). 1015, **Reaction between sodium phosphates and calcium hydroxide** (Hayashi and Matsui). 1016, **Behaviour of sulphurous acid towards thiosulphuric acid** (Foerster and Vogel). 1017, **Determination of halides** (Berg); **Determination of halogen compounds** (König). 1018, **Electrometric titration of phosphates** (Bodforss); **Titration of hypophosphorous acid and its salts** (Kolthoff). 1019, **Determination of ferro- and ferri-cyanides** (Cumming). 1021, **Potash analysis apparatus** (Müller).

Patents.

System of concentrating sulphuric acid. A. A. Vineyard (U.S.P. 1,596,907, 24.8.26. Appl., 9.9.25).—The acid flows from the heater into a chamber fitted with an agitator, by which means the organic matter is broken up into small particles and the released vapours and gases pass through a condenser. The acid from the chamber outlet then flows through a second heater, and through a series of concentrating stills, each having an exit leading to the condenser. H. Royal-Dawson.

Chemical analysis apparatus and process [for oleum]. H. Howard, Assr. to Grasselli Chemical Co. (U.S.P. 1,594,264, 27.7.26. Appl., 1.3.24).—Standard acid and samples of oleum are passed through a temperature equaliser and then mixed in equal quantities

in the analysis chamber (cf. J.S.C.I., 1910, 3). The resulting change in temperature actuates a heat-sensitive device, *e.g.*, an electrical resistance thermometer, placed in the analysis chamber, and the effect is recorded on a suitable scale as percentage of free sulphur trioxide. The heat-sensitive device can also control the absorbing acid supply valve. E. S. Kreis.

Concentrating and purifying phosphoric acid. H. E. La Bour (U.S.P. 1,597,984, 31.8.26. Appl., 13.9.23).—The vapour evolved from phosphoric acid at a temperature below its b.p. is blown away, carrying the fluorine with it. H. Royal-Dawson.

Manufacture of hydrofluoric acid. M. Buchner (E.P. 255,464, 2.6.25. Conv., 2.6.24. Addn. to 234,852, B., 1925, 670).—A volatile fluoride, such as boron fluoride, is added to a mixture of hydrofluoric and hydrofluosilicic acids, and the latter, together with the complex hydrofluoric acid produced, are caused to react with a metal fluoride, whereby hydrofluoric acid and the metal salts of hydrofluosilicic acid and the complex hydrofluoric acid are obtained. These, when heated, yield the metal fluoride, silicon fluoride, and the volatile fluoride first added. H. Royal-Dawson.

Apparatus for drying and neutralising ammonium sulphate etc. T. B. Smith (E.P. 256,385, 13.7.25). —The apparatus consists of a shallow steam-heated pan, in which are one or more sets of stirring blades mounted on a radially disposed moving arm operating from a main shaft, and provided with a counterbalance lever worked by hand or trip mechanism. In action, one set of blades is put in operation to convey the material from the periphery of the pan to the centre, followed by the second set in conjunction with the first to convey material from the centre to the periphery, the first set being finally thrown out of action to allow the second set to convey the material to the delivery passage. H. Royal-Dawson.

Catalytic oxidation of ammonia. I. G. Farbenind. A.-G., Assees. of O. Balz and F. Reuscher (G.P. 431,506, 23.12.24).—Oxygen, or gases rich in oxygen, and ammonia are circulated through one or more catalyst chambers, ammonia being added as necessary to avoid an explosive mixture. A portion of the reaction gas is drawn off, continuously or intermittently, for absorption. W. G. Carey.

Production of alkali carbonates from alkali sulphates. W. H. Friedrich (G.P. 431,509, 17.9.24).—An alkali carbonate is produced by the interaction of a freshly precipitated alkaline-earth carbonate, particularly barium carbonate, and a solution of an alkali sulphate with the addition of a small amount of an ammonium salt. W. G. Carey.

Preparation of stable alkaline-earth polysulphide solutions. E. Glücksmann (G.P. 431,642, 17.4.25).—The solutions are stabilised in a similar manner to alkali polysulphide solutions (see G.P. 419,910; B., 1926, 237). W. G. Carey.

Manufacture of alkali hydrosulphites [hyposulphites]. Farbenfabr. vorm. F. Bayer & Co. (E.P. 247,524, 12.12.25. Conv., 14.2.25).—In the manufacture of hyposulphites from alkali bisulphites and alkali amalgam, the alkali sulphite in the reaction liquid is maintained at not more than 45 g. per litre by withdrawing the mother liquor and replacing it by water and salt. H. Royal-Dawson.

Production of barium oxide of high porosity and purity. H. Schulze (G.P. 431,617, 11.6.24).—A mixture of barium carbonate and charcoal is heated indirectly, by electrical means, in such a manner that the mass is heated from two or more sides. The ordinary or slightly increased pressure may be used. W. G. Carey.

Separation of barium chloride from solution. Rhenania Verein Chem. Fabr. A.-G., Assees. of F. Rüsberg (G.P. 431,643, 2.10.24; see G.P. 429,716; B., 1926, 778).—Rock salt is used in place of common salt, and is mixed by degrees with definite portions of the solution, the impurities being removed by filtration, after each introduction of rock salt. The rock salt may partly be replaced by pure salt. W. G. Carey.

Production of magnesium carbonate with simultaneous recovery of ammonium salts. B. Waeser (G.P. 431,618, 24.2.23).—Solutions of magnesium salts, or crude potash salts containing magnesium, are treated with not more than 10% of ammonia, and the mixture is cooled with the addition of carbon dioxide so that the formation of bicarbonate is avoided. The precipitate of magnesium carbonate is separated by filtration from the solution of ammonium salts with any potassium salts. W. G. Carey.

Process for producing stannous chloride. W. T. Little, Assr. to Metal & Thermit Corp. (U.S.P. 1,597,653, 24.8.26. Appl., 12.1.25).—A solution of stannic chloride is reduced in the cathode compartment of a divided cell under conditions preventing the deposition of tin. H. Royal-Dawson.

Production of crystallised sulphates of iron, copper, zinc, and nickel. G. Agde (G.P. 431,581, 14.1.25).—Sufficient sulphuric acid is added to the solution so that it may be used again as dissolving acid, the saturated sulphate solution is cooled, and such conditions of temperature and acid and salt concentration are maintained as to prevent the contamination of the crystals by simultaneous formation of crystals with less water. W. G. Carey.

Treating vanadium ores and solutions. W. E. Stokes, Assr. to U.S. Processes Co. Inc. (U.S.P. 1,597,216, 24.8.26. Appl., 19.1.21).—Lead vanadate, precipitated by the addition of a lead salt to a leach solution containing vanadium compounds, is separated and decomposed by excess of acid in a salt solution in which the resulting vanadium oxide is soluble. The insoluble lead salt is filtered off and vanadium oxide is precipitated by reducing the acidity of the solution. S. S. Woolf.

Production of pure sulphides. Ges. f. Chem. & Hüttenwesen (G.P. 431,253, 3.4.24).—A solution of an alkaline-earth sulphide is caused to react with an alkali carbonate solution under pressure, the sulphide liquor being concentrated *in vacuo*, in stages. W. G. Carey.

Production of sodium aurothiosulphate. I. G. Farbenind. A.-G., Assees. of O. Ripke (G.P. 431,580,

6.5.25).—A solution of a gold salt and sodium thiosulphate is evaporated under reduced pressure in a neutral atmosphere. W. G. CAREY.

Production of silicon carbide from silicic acid and charcoal. A. REITZ (G.P. 431,759, 13.3.25).—Crude fluorine compounds are added as catalysts, the volatilisation of which is prevented by surrounding the furnace by a casing. W. G. CAREY.

Extracting iodine and bromine from solutions. G. VELLARDI, and A.P.I.C.E. SOC. AN PROD. ITALIANI CHIMICI ESTRATTIVI (E.P. 257,134, 18.11.25).—Solutions or water containing the halogens in the natural state, or in residual mother-liquors, are admitted under pressure together with a reagent to liberate the halogen and a solvent of iodine and bromine (*e.g.* petroleum) into an emulsifier, the liquid mass then passing into centrifugal machines or decanting tanks to separate the solvent. The latter is then passed into a second emulsifier, where it meets a solution of a substance capable of converting the halogens into soluble salts, and the resulting solution is finally separated from the solvent by decantation. H. ROYAL-DAWSON.

Production of colloidal sulphur. I.G. FARBENIND. A.-G., Assees. of F. WINKLER and F. GILLER (G.P. 431,505, 6.9.25).—Polysulphide solutions, with the addition of protective colloids, are sprayed into vapour or gas, with or without the addition of an electrolyte. W. G. CAREY.

Manufacture of graphite. A.-G. F. ANILIN-FABR. (E.P. 252,662, 12.12.25. Conv., 26.5.25).—Carbon is heated (*e.g.*, in the electric arc) in the presence of air or other suitable gas at or above 3900°, and the escaping vapours are condensed on a highly heated surface in the immediate neighbourhood of the heated material, having a temperature of at least 1500°. H. ROYAL-DAWSON.

Purification of hydrogen [for making synthetic ammonia]. L'AIR LIQUIDE, SOC. ANON. POUR L'ÉTUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (E.P. 250,520, 26.5.25. Conv., 9.4.25. Addn. to 238,175, cf. B., 1925, 872).—In purifying hydrogen for the manufacture of synthetic ammonia, liquid air (in place of liquid nitrogen) is introduced in small quantities into the expanded hydrogen, and the oxygen is removed prior to the actual synthesis by reaction with the hydrogen in the mixture in presence of a catalyst. H. ROYAL-DAWSON.

Catalytic preparation of hydrogen and phosphoric acid. I. G. FARBENIND. A.-G., Assees. of A. MITTASCH and G. WIETZEL (G.P. 431,504, 23.11.24).—Water vapour, with or without oxygen or gases containing oxygen, acts upon phosphorus vapour, charcoal being employed as catalyst. W. G. CAREY.

Separation of helium from gas mixtures. F. PANETH and K. PETERS (G.P. 431,507, 2.12.25).—Gases containing helium are made to impinge upon a heated, gastight diaphragm, *e.g.*, of glass. At a high temperature the helium diffuses through more quickly than the heavier gases. The diaphragm may be heated by an electrical resistance or by hot gases, and the gas pressure on both sides may be varied at will. W. G. CAREY.

Electrolytic decomposition of chlorides. E. SCHLUMBERGER, Assr. to KOHOLYT A.-G. (U.S.P. 1,598,018, 31.8.26. Appl., 17.9.25. Conv., 28.7.24).—See E.P. 254,163; B., 1926, 757.

See also pages 908, **Hydrogen** (G.P. 431,758). 922, **Alkali metals** (U.S.P. 1,597,231). 926, **Calcium nitrate** (E.P. 242,990). 934, **Chlorine water** (G.P. 429,037).

VIII.—GLASS; CERAMICS.

[Glass] tank block corrosion by shelving. F. C. FLINT and A. R. PAYNE (J. Amer. Ceram. Soc., 1926, **9**, 613—617).—Part of the rapid dissolution of tank blocks by glass is due to attack in an upward direction from cracks in the surface of the block. The upward penetration is probably due to the formation of a ledge at the crack upon which the more saturated and viscous glass rests. For this reason the ledge is attacked less rapidly than the roof of the crack, which is exposed to fresh glass. Density measurements of glass against the wall of the tank, as well as analysis of small clay–glass melts, proves the increase of sp. gr. with increasing solution of block. A. COUSEN.

Temperature of pyrex and porcelain in sunlight. J. T. LITTLETON, JUN., and W. W. SHAVER (J. Amer. Ceram. Soc., 1926, **9**, 618—625).—Calculation shows the maximum surface temperature in sunlight (with shade temperature 40°) of porcelain to be 93°, of pyrex glass 46·5°. With a shade temperature of 22°, small plates of porcelain increase in sunlight 19·7° in ½ hr., pyrex plates remaining unchanged. When placed at the focus of metal reflectors in intensified sunlight the temperature increase of the porcelain is 3·5 times that of the pyrex. A. COUSEN.

Requirements of refractories for manufactured gas plants. S. S. COLE (J. Amer. Ceram. Soc., 1926, **9**, 462—473).—The requisite qualities, and the service expected, of the refractories used in four types of installations, viz., gas producers or water-gas plants, horizontal and vertical retorts, and gas ovens, are discussed in broad outline. F. SALT.

Effect of repeated burning on the structure and properties of lime-bonded silica bricks. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1926, **25**, 82—93).—A number of lime-bonded bricks made from Sheffield ganister were subjected to 11 reburnings in an industrial kiln, samples being withdrawn for examination after each reburn. The bricks were tested for powder density, volume changes, mechanical strength, and changes in structure. The powder density tests showed that the major part of the quartz inversion was accomplished in the first burn, but the proportion of unchanged quartz decreased steadily until the 7th burn. Very little change in volume occurred after the first burn. Porosity decreased steadily down to the 7th burn, after which it remained fairly constant. A marked increase in crushing strength up to the 7th burn was noticed, and there was a steady increase in the transverse strength up to the 5th burn. The results tend to emphasise the importance of the "soaking" period during firing. No change in chemical composition took place as the

result of reburning. Changes in structure are illustrated by photomicrographs. F. SALT.

Fusion points of firebrick–coal ash mixtures. L. C. HEWITT (J. Amer. Ceram. Soc., 1926, 9, 575—582).—Cone fusion points of mixtures of samples of six firebricks with each of five samples of coal ash over the range 10—40% of brick indicated the presence of eutectics in the mixtures. The effect of reducing conditions upon the results was very marked. Coal ashes rich in iron did not give the most pronounced effects. No definite grouping of the firebricks could be made from the results. A. COUSEN.

Comparison of the uniformity of strength and texture of firebrick made by different processes. A. E. R. WESTMAN and W. H. PFEIFFER (J. Amer. Ceram. Soc., 1926, 9, 626—632).—The following order of decreasing uniformity of transverse strength of firebricks was found :—(1) Semi-dry press, (2) stiff mud repress, (3) dry press, (4) hand-moulded. The order of decreasing uniformity of texture was 1, 3, 4, 2. A. COUSEN.

Some successful cone 10 saggar bodies. E. H. FRITZ (J. Amer. Ceram. Soc., 1926, 9, 606—612).—A successful saggar body for firing at cone 10 must have a dry modulus of rupture not less than 80 lb./sq. inch, an absorption at cone 10 of not less than 8% nor more than 11½%, and a shrinkage of not more than 7%. Mixtures of clays giving the best results were (*a*) Maryland No. 1 10%, N.J. No. 3 15%, N.J. No. 4 25%, grog 50%, and (*b*) Ohio No. 2 38%, Penn. No. 2 17%, grog 45%, with the grog size 75% of 34—12-mesh and 25% 2—3-mesh. A. COUSEN.

Thermal expansion of saggar clays. R. F. GELLER and R. A. HEINDL (J. Amer. Ceram. Soc., 1926, 9, 556—574).—The thermal expansion of 49 clays was determined up to 1000°, by heating bars (50% raw clay, 50% grog) previously fired at 1230° in a vertical electric tube furnace. Expansions were transmitted to a gauge by a quartz rod. In addition, samples were air-quenched at successively increasing temperatures until fracture occurred. Two types of clay were distinguished, one expanding fairly uniformly to 500°, the second giving a decided increase in the rate between 100° and 200°. In the quenching test practically all clays of the first group exceeded 500° before cracking, the majority of the second group failed below 500°. Repeated firings tended to decrease the expansion between 500° and 600°, and increase that between 100° and 200°. Artificial corundum increased the total average expansion of clays with which it was used, and tended to eliminate irregular expansion due to inversion of free silica. A. COUSEN.

Use of Georgia and North Carolina kaolins in a semi-porcelain body. S. E. HEMSTEGER and W. C. STIEF (J. Amer. Ceram. Soc., 1926, 9, 444—445).—A comparative study with these clays and two English china clays when used in a semi-porcelain body for dinnerware indicated that the American clays were adapted to this purpose, provided that the necessary readjustments were made in substituting for the English clays. F. SALT.

Enamel clays. V. W. BOEKER (J. Amer. Ceram. Soc., 1926, 9, 399—411).—Twelve typical enamel clays were examined for water of plasticity, drying shrinkage, modulus of rupture, and behaviour on firing to 1000°. Enamel slips were prepared by mixing 10 pts. of clay with 100 pts. of ordinary commercial frits. The viscosity, the ability to hold the frit in suspension, and the hydrogen-ion concentration of the slips were determined. The fineness of the clays was tested on a 300-mesh sieve, and they were subjected to petrographic and chemical analysis. Finally, the clays were tested in a series of cover coats on sheet steel, the enamels being made up of 100 pts. of frit, 7 pts. of clay, 4 pts. of tin oxide, 3 pts. of "leukonin," and about 40 pts. of water. The fired test pieces were examined by the impact test and the thermal shock test. The suspension test did not give reliable data, and the results did not agree with the hydrogen-ion concentration. All clays having p_H values greater than 6·0 proved to be good suspension agents for the cover coat enamels. Impurities in the clays affected the results more by their nature than their quantity. Small amounts of carbonaceous matter, other than coal or lignite, had no effect on the enamel, but lime, if present in excess of 1%, caused blistering. The degree of maturity and the lustre were inversely proportional to the porosities of the clays at 1000°. The resistance of the fired enamels to impact and thermal shock was influenced greatly by the clay used, but no direct relation was found between these factors. F. SALT.

Practical chemical control of [enamel] pickling room solutions. B. T. SWEELY (J. Amer. Ceram. Soc., 1926, 9, 590—592).—The acid solution used for pickling in the enamel industry is titrated by adding to 10 c.c. in a special cylinder sufficient $\bar{N}/2$ caustic soda (containing methyl-orange) to neutralise, and reading the strength of acid from graduations on the cylinder. Similar methods are used for the cleaning and neutralising solutions. A. COUSEN.

Determination of relative surface areas of powders. KOEHLER.—See I.

PATENTS.

Process and apparatus for purifying clay etc. F. PARENTANI (E.P. 255,300, 2.12.25).—Colouring impurities are removed from clays and similar materials by treating them in the dry state with hydrogen sulphide at atmospheric temperature. The material may first be subjected to the action of a vacuum. It is finally treated with an acid solution (hydrochloric acid) to dissolve the sulphides formed, the hydrogen sulphide produced being used to treat further material. F. SALT.

Removal of iron from clays, bauxites, etc. H. FLEISSNER (Austrian P. 102,553, 7.8.23).—Before being treated with dilute acids, clays are subjected to the action of hydrogen sulphide to remove the iron. The iron sulphide formed is best decomposed with sulphur dioxide or with gases containing it. F. SALT.

Manufacture of refractory bricks. PFÄLZISCHE CHAMOTTE- U. THONWERKE (SCHIFFER & KIRCHER) A.-G., Assees. of G. WAGAPOFF (E.P. 248,369, 22.2.26. Conv., 25.2.25).—The refractory clay is worked up without

the addition of grog, and with a moisture content not exceeding 20% by weight. It is disintegrated without previous drying or heating or subsequent moistening to a grain size varying from a fine powder to 10 mm. The material is then intimately mixed, moulded under pressure, and fired in the usual way. F. Salt.

Manufacture of durable earthenware. H. Oexmann (E.P. 231,469, 14.3.25).—The body mixture, or the roughly-shaped article, is subjected to high pressure (300 atm.) through a gaseous, liquid, or powdered medium. The pressed article may be turned, and is then fired. F. Salt.

Muffle kilns [for ceramics etc.]. S. Crosbie and C. H. F. Collard (E.P. 253,959, 21.9.25).—An "endless" series of separate, continuous muffle kilns connected by flues is arranged polygonally around a central space. Each muffle in turn becomes partly heated by waste gases from the kiln immediately preceding it. A combustion chamber below the floor of each kiln is connected with a number of flues formed in the surrounding kiln walls and with a single crown flue eading to down flues in the walls. F. Salt.

[Tunnel] kilns or ovens for baking pottery etc. R. M. d'Arleux and E. Violette (E.P. 254,023, 28.3.25).—A mixture of gaseous fuel and air is introduced into the tunnel through tuyères constructed with a thermosiphonic circulatory cooling system. The size and number of the tuyères, the pressure of the gaseous fuel, and the capacity of the hearth are so adjusted that the pressure in the kiln is greater than atmospheric. Sloping baffles are fitted at the mouths of the tuyères to spread the flames. F. Salt.

[Brick or pottery] kilns. G. V. Evers (E.P. 254,438, 24.4.25).—In a multi-chamber kiln, fire boxes are provided at each of the four corners of each chamber except the end chambers, in which the furnaces are arranged in the end walls. A low baffle wall is built near the inner end of each furnace to give a short vertical passage for the hot gases. Passages are provided at the base of the partitions between the chambers to give inter-communication. A central outlet in the floor of each chamber leads to the chimney flue. F. Salt.

[Down-draught] kilns for the manufacture of bricks and pipes. E. Evans (E.P. 255,188, 18.5.25).—The hot gases are drawn through the perforated floor of the kiln chamber and pass through hooded pipes covering holes in an intermediate floor spaced from the kiln base. The holes near the peripheral wall in the intermediate floor are of larger cross-section than those near the centre. A portion of the gases passes from the space beneath the intermediate floor direct to the chimney, the remainder being drawn through closed passages beneath the kiln base, which communicate directly with the space beneath the larger apertures in the second floor. F. Salt.

Enamelling metal articles. M. Auger (E.P. 254,452, 5.5.25).—To produce a surface to which vitreous enamels will adhere on brass and similar yellow metal alloys, without the application of a copper coating, the alloy is treated with an acid (nitric acid, or a mixture of nitric and sulphuric acids, and/or a pickle (sodium chloride) to dissolve the metals, with the exception of the copper, from the surface. A practically pure copper surface is thus produced. F. Salt.

Artificial stone for abrasive and other purposes. H. Wade. From Carborundum Co. (E.P. 257,391, 23.7.25).—See U.S.P. 1,553,105; B., 1925, 882.

Machinery for manufacturing glass-ware. P. C. Rushen. From Hartford-Empire Co. (E.P. 257,637, 7.3.25).

Kilns (U.S.P. 1,590,462 and 1,590,935).—See I.

IX.—BUILDING MATERIALS.

See A., Oct., 1012, **Activation of inert varieties of calcium sulphate** (Budnikov).

Patents.

Rotary furnaces for burning cement and the like. A. Schmid (E.P. 253,819, 4.1.26).—The discharge head of a rotary furnace is constructed at its lower end as a cooling chamber, to which air is admitted under pressure. The air passes from the cooling chamber through the material and is then utilised for combustion purposes. In an alternative construction, the head widens out at the lower end to form a hopper-shaped cooler, within which is a cone having a louvre-like upper part. The burnt material falls in a thin layer over the cone. Air under pressure is introduced into the cone and passes through the louvre opening over the material. F. Salt.

Production of hydraulic cement from oil shale and limestone. Rekord Cement Ind. G.m.b.H., Assees. of O. Tetens (G.P. 429,651, 10.12.22).—The burnt material (cf. U.S.P. 1,536,165; B., 1925, 549) is quenched in a closed vessel with the ammoniacal liquor obtained in the process. W. G. Carey.

Cement and process of making it. E. C. Eckel (U.S.P. 1,591,662, 6.7.26. Appl., 31.3.26).—A mixture containing alumina, iron, silica, and lime is fused in the presence of sufficient free carbon to form ferrosilicon. F. Salt.

Mixture for use as a cement cold-glaze. J. Langbein (E.P. 247,947, 11.2.26. Conv., 18.2.25).—A mixture to be added to the usual cement mixture for cold-glazes contains 250 pts. by weight of polymerised linseed oil, 625 pts. of ammonia water, 6000 pts. of lime water, 1000 pts. of infusorial earth, which may be admixed with asbestos, 625 pts. of barium or strontium oxide, or 1500 pts. of a chromate. F. Salt.

Process of impregnating wood. K. Bubla (U.S.P. 1,597,010, 24.8.26. Appl., 20.6.24).—Wood is impregnated under reduced pressure with a dilute solution of a suitable substance, followed by one of higher strength on again reducing the pressure. H. Royal-Dawson.

Manufacture of aluminous cements. E. Martin (E.P. 251,618, 20.4.26. Conv., 30.4.25).—See F.P. 597,978; B., 1926, 409.

Paving and surfacing material. C. E. Ramsden (U.S.P. 1,598,505, 31.8.26. Appl., 19.8.25. Conv., 1.8.24).—See E.P. 243,418; B., 1926, 129.

Prepared tar for road-making (E.P. 257,024).—See III.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of oxidation reactions on the blast-furnace process. F. Wüst (Stahl u. Eisen, 46, 1213—1221).—A comparison is made between iron furnaces for ore-reduction and refining furnaces. In both, the solid phase has a reducing action, whilst the gas phase acts as a reducing agent in the former and as an oxidising agent in the latter. The predominating influence of the gas phase is emphasised. In the blast furnace, an oxidising zone is present in front of the tuyères, causing secondary reactions which impair the quality of the product and reduce the economic efficiency of the process. The proportions of carbon monoxide and dioxide and of oxygen at different distances from the tuyères are shown, and from these data, the extent of the oxidising zone with variation in hearth diameter is determined. The effect of hearth diameter on the quantity of blast required, coke consumption, and yield of iron is shown. Tuyères entering the furnace tangentially are advocated. L. M. Clark.

[Formation of] graphite scum in cast iron. B. Osann (Stahl u. Eisen, 1926, 46, 1320—1324).—The formation of a scummy porous mass of iron, very rich in graphite, during the casting of iron, is due to the precipitation of graphite in the liquid iron during cooling, but before solidification. This scum rises to the top of the casting and prevents proper filling of the mould. The presence of large quantities of sulphur or silicon, which tend to reduce the solubility of carbon in molten iron, favours the formation of this scum. Examples from practice are given with details of the melting conditions which gave rise to this behaviour in each case. To avoid the separation of graphite from the liquid mass the carbon content of the metal should not rise too high, and preheated moulds should be used for casting, so as to mitigate the bad effects of rapid cooling. In regulating the carbon content of the charge for the cupola, attention must be paid to the undoubted effect of the nature and quantity of the slag on the composition of the metal. The theories put forward to account for the separation of graphite from liquid iron are briefly discussed. A. R. Powell.

Recrystallisation temperatures of cold-rolled electrolytic iron and open-hearth steel strip. J. R. Freeman (Trans. Amer. Soc. Steel Treat., 1926, 10, 67—86).—Two samples of open-hearth steel strip and one sample of electrolytic iron, 0·065 in. thick, were annealed and then cold-rolled, in one experiment with a 90% reduction in six passes, and in a second experiment with a 73% reduction in two passes. Specimens were then annealed at various increasing temperatures. There was a marked decrease in strength and increase in elongation between 500° and 600° in the case of the steel strip, and between 400° and 500° for the electrolytic iron. In the latter case the tensile strength at 500° was lower the greater the preliminary amount of cold working. Photomicrographs show that cold-rolling distorted the grain structure but caused no recrystallisation. On annealing, the degree of grain refinement increased with the amount of cold work. Recrystallisation commenced between 500° and 600° for the steel strip and between 450° and 500° for the electrolytic iron, the lower recrystallisation temperature being due to its relatively higher purity. T. H. Burnham.

Influence of nitrogen on the solubility of ferrous materials in hydrochloric acid. H. H. Gray (J.S.C.I., 1926, 45, 365—366 T).—A sample of mild steel is rendered almost insoluble in hydrochloric acid of varying strengths, up to 20% by weight, by being heated in nitrogen for about 6 hrs. at 1000°. The loss of weight by the nitrogenised steel due to attack by hydrochloric acid is 0·68%, as compared with a loss of 46% suffered by a duplicate sample not treated with nitrogen.

Macroscopical examination of iron and steel. F. P. Gilligan and J. J. Curran (Trans. Amer. Soc. Steel Treat., 1926, 10, 9—30).—The progressive nature of the solidification of steel and the influence of the diffusible and less diffusible elements and of sonims on the macrostructure are described. Of the various macro-tests the hot acid etch is considered the most productive of information. A solution of hydrochloric acid or of hydrochloric and sulphuric acids is preferred. Photographs are given illustrating the use of hot acid etching in demonstrating the following phenomena in steel:—segregation, laps, ingot structure in castings, the persistence of dendritic structure in forgings, the flow of metal in forgings, decarbonised surface layers in tool steel, incipient fatigue cracks, and localisation of hardening stresses by deep stamping. The process may be used to determine the method by which steel balls are forged. The indications of hot acid etching are corroborated by photomicrographs and analytical data. T. H. Burnham.

Dendritic structure and crystal formation [in steel]. B. Stoughton and F. J. G. Duck (Trans. Amer. Soc. Steel Treat., 1926, 10, 31—52).—Overheated 1·28% carbon steel showed an intercrystalline fracture under shock. It is suggested that this was due to large crystals and consequent large surfaces of intercystalline cement. The same reason is advanced for intercrystalline fracture at high temperatures. Photomicrographs show that although the crystals of pearlite were large, the individual crystals of ferrite and cementite were the same size as in 1·16% C file steel. Small crystals and interlamellar crystals throughout the eutectoid area were formed by pressure due to the austenite–pearlite expansion. At a magnification of 4800 the cementite crystals were seen to be distorted and to tend to thicken and fold back on themselves. The Brinell hardness was lower the larger the crystals. In the discussion it was brought out that the intercrystalline fracture might be due to large spines of cementite along the grain boundaries, or to high concentration of impurities along the boundaries of the crystals, rather than connected with the amorphous intercrystalline layer, also that the large crystals formed at high temperature are different from the large primary crystals under discussion. T. H. Burnham.

Influence of heat treatment and carbon content on the structure of pure iron–carbon alloys. W. L. Fink and E. D. Campbell (Trans. Amer. Soc. Steel Treat., 1926, 9, 717—754).—*X*-Ray studies were made on two series of carbon steels prepared from Armco iron

using the Bohlin and wedge methods. The first series, containing up to 1·30% C, was oil-hardened, and the second, containing up to 1·50% C, was water-hardened and tempered. In the oil-quenched specimens, the lattice was much the same as for pure α-iron, but there were indications that some parts of the lattice slightly dilated with increasing carbon content. Weak γ-lines did not appear until 0·97% C, the lattice constant a for γ-iron being 3·56 Å. In the water-quenched specimens up to 0·21% C the α-lattice was not deformed, but at higher contents a part was dilated until at 0·43% C very little of the lattice retained its original dimensions. γ-Iron lines appeared at 0·66% C, and at a somewhat higher content a new crystal structure, viz., a body-centred tetragonal lattice, made its appearance, the amount present increasing with the carbon content and attaining uniform dimensions at 1·50% C ($a = 2·85$, $c = 3·02$ Å). At 0·9% C for the γ-iron a was 3·59 Å, and for higher carbon contents it dilated to 3·61 Å. On tempering, the changes were in the reverse sense. At 100° the body-centred tetragonal lattice disappeared, and at 203° the γ-iron lines disappeared. The α-lattice assumed the dimensions for pure iron as the temperature increased. Iron carbides did not give any carbide lines, although precipitated in sufficiently large particles to be visible at 500 magnifications. The results from the oil-hardened specimens were the same as for water-quenched and tempered a little below 200°. T. H. BURNHAM.

Effect of reheating on cold-drawn [steel] bars. S. C. SPALDING (Trans. Amer. Soc. Steel Treat., 1926, 9, 685—716).—Rods of three types of steel, containing respectively about 3% Ni, 0·77—1·44% Mn, and 0·96% Cr and 0·18% V, were cold-drawn to a finished size of 0·45 in. with 42·5, 28·1, and 18·9% reductions, and then reheated to various increasing temperatures. After reheating to 315° the steels gained considerably in yield point with only slight reduction in tensile strength, ductility and impact values. Above 600° for the manganese and nickel steels, and 650° for the chromium–vanadium steel the yield point and tensile strength fell rapidly and the ductility and impact test increased. The response of hot-rolled and cold-rolled bars to reheating was practically identical. The Izod figures for cold-drawn bars were higher than for oil-hardened bars at low reheating temperatures, but above 315° the values for the heat-treated bars rose rapidly; for cold-drawn bars the rise did not occur till 600—650°. The chromium–vanadium steel had the highest impact value for a yield point under 50 tons per sq. in.; above this value it was equalled by the nickel steel. The minimum Izod value was found on unquenched cold-rolled steel at about 315° reheating temperature. This indicated that the same phenomenon found in quenched and tempered samples was not due to an austenite–martensite transformation. The electrical conductivity of the manganese and chromium–vanadium steels decreased slowly to 425°, and then fell rapidly; that of the nickel steel decreased more uniformly. The effect of cold work on the conductivity was practically nil. T. H. BURNHAM.

Distribution of hardness in quenched carbon steels, and quenching cracks. T. KASÉ (Sci. Rep. Tôhoku, 1926, 15, 371—386).—Cubes of Swedish carbon steel were heated in an electric furnace to a desired temperature and quenched after 10 min. by immersing in an oil- or water-bath. After polishing, the hardness of different points of the surface was measured by the Shore scleroscope. Quenching causes the hardness to increase to about three times that of the annealed specimens, and in severe quenching the periphery is always less hard than the interior, the difference increasing with rise of quenching temperature. The reverse is true in the case of soft quenching. This is explained by the presence of retained austenite in martensite. The change of the retained austenite into martensite explains the considerable increase in hardness by cooling in liquid air, and this change also causes the hardness of a specimen to increase with age. Specimens quenched in water from above 900° always cracked, the lines of fracture cutting the equi-hardness curves orthogonally. Samples containing less than 0·9% of carbon did not crack. The cracking is not caused by a thermal stress, but is due to the difference between the specific volumes of austenite and martensite. E. S. HEDGES.

Specific heat of carbon steels. S. UMINO (Sci. Rep. Tôhoku, 1926, 15, 331—369).—The specific heats of steels with carbon contents 0·09—2·84% were determined by the method of mixtures over the temperature range 23—1250°. The specific heat increases with rise of temperature up to the A3 transformation point, above which it is constant. The mean specific heat of cementite increases from 0·149 at 150° to 0·220 at 850°. For the dissolution of 1 g. of carbon in iron 1760 cal. are required, and 16·1 cal. for the dissolution of 1 g. of pearlite in iron. The heat changes for the dissolution of pearlite in iron, the transformation of martensite to pearlite and of austenite to martensite, were found to be proportional to the carbon content. The heat of transformation from austenite to martensite for a eutectoid steel is 5·9 cal. The A1 transformation is a function of temperature and time, but the A2 transformation is a function of temperature only. E. S. HEDGES.

Optimum temperature for the extrusion of (α+β) brass. W. SCHREITER (Z. Metallk., 1926, 18, 285—287).—Extrusion experiments on brasses containing 52—66% Cu show that the optimum temperature for the extrusion varies with the composition of the alloy, but is, in all cases, just slightly above the temperature at which the whole of the metal is in the β form. Thus, brass containing 61·5% Cu consists entirely of β above 758°; if extruded into tubes at 740° it has a tensile strength of 40 kg./mm.² with an elongation of 32%, at 750° these values are 42·7 and 33·7, and at 760° 43 and 41 respectively. The microstructure of the metal extruded at 740° consists of long parallel lines of alternating α and β, whereas at 760° it consists entirely of the characteristic coarse-grained crystal structure usually obtained after pressure. Annealing at 760° of this brass extruded below 758° produces the satisfactory β-structure. Extrusion of the brass at 770—780° causes, however, a much more coarsely crystalline structure to develop, and the metal becomes very weak, often fracturing during extrusion or on subsequent cooling. Similar characteristics hold

for other brasses containing 52—66% Cu, so that in all cases the metal in the press should be maintained as near as possible to the temperature at which the $\alpha + \beta \rightarrow \beta$ change is completed, allowance being made for the rise in temperature caused by the pressure required for the extrusion. A. R. Powell.

Determination of tin in cassiterite. A. Pirlot (Bull. Féd. Ind. chim. Belg., 1926, **5**, 281—284).—The finely ground sample is digested with hydrochloric acid to remove iron and the residue is collected, washed, dried, ignited, and finally reduced to metal by heating for 2 hrs. at 740° in a current of hydrogen. The loss in weight during the last-named treatment is a measure of the tin content of the ore. As a check the metal is dissolved in hydrochloric acid, the insoluble material separated, again reduced in hydrogen and any further metal extracted as before, and the combined stannous chloride filtrates are neutralised with ammonia, oxidised with iodine, and boiled with sodium sulphate or ammonium nitrate. The precipitated metastannic acid is collected, washed with ammonium nitrate solution, and ignited to stannic oxide. For a good sample of cassiterite the tin content calculated from the loss on reduction agrees very closely with that obtained by weighing the tin oxide. A. R. Powell.

Oxidising salt test and the intercrystalline corrosion of aluminium and its alloys. H. Biegler (Z. Metallk., 1926, **18**, 288—289).—The action of the oxidising salt solution of Mylius (B., 1925, 552) on aluminium takes place chiefly along the boundaries of the crystal grains and, if carried out at ordinary temperatures over a period of several days (renewing the salt solution every day), valuable information on the intercrystalline corrosion of aluminium and its alloys is obtained. In the case of technical aluminium (98—99% Al), the loss of metal per unit area increases slowly but steadily with the time provided that the original surface of the metal produced by rolling remains untouched; if this skin is removed by etching, rapid attack of the metal takes place during the first two days, after which a new protective skin is formed and the rate of corrosion falls to the normal. In the case of aluminium alloys the protective action of the natural skin is more marked, but in both the etched and unetched alloys corrosion takes place much more readily than in the case of aluminium itself, the rate rising with the time to a maximum and then decreasing rapidly as a protective film forms. A. R. Powell.

Stress-strain curves and physical properties of metal, with particular reference to hardness. H. P. Hollnagel (Trans. Amer. Soc. Steel Treat., 1926, **10**, 87—108).—The stress–strain curve is interpreted in terms of atomic restitutional forces and slip whether in mass or of an atomic nature. Deformation may be either elastic, hysteretic, or permanent, or all combined in a random aggregate of crystals under stress. Various physical properties of metals, *e.g.*, brittleness, ductility, resilience, toughness, elasticity, and rigidity, are interpreted in terms of the characteristic points of the stress–strain curve. Hardness is considered an average property which includes the effects of all other characteristics of the stress–strain curve; it may be defined as the work done per unit volume per unit strain, and has the physical dimensions of pressure. The lack of correlation between indentation, scratch, and rebound hardness determinations is examined. Theoretically the new definition makes possible the unification of all hardness measurements. Indentation hardness depends on surface hardness (proportional to the elastic limit) and the mechanical properties which determine cold work. T. H. Burnham.

See also A., Oct., 997, **Distortion of iron crystals** (Taylor and Elam); **Pure zinc** (Freeman and others); **Behaviour of single crystals of aluminium under stress** (Gough and others); **Deformation of tungsten crystals** (Smithells and others). 999, **Density and expansion of white and grey pig-iron** (Sauerwald and Widawski). 1001, **Thermal anomalies in solid solutions** (Chevenard). 1016, **Precipitation of metals from non-aqueous solutions** (Müller and others).

Refractory articles from tungsten powder. Hardén.—See XI.

Patents.

Electrolytic production of an adherent burnish on rustless iron or steel. W. A. F. Pfanhauser, and Langbein-Pfanhauser-Werke A.-G. (E.P. 257,140, 30.11.25).—A superficial layer of material is removed from the rustless alloy to allow subsequent deposition of a coating of iron. The process is carried out by making the alloy the anode in a bath of neutral iron salts with some neutral conducting salts, followed by reversal of the current. The deposit of iron is oxidised to produce the burnish. L. M. Clark.

Removal of arsenic from tin. H. Harris (E.P. 257,023, 20.5.25).—Molten arsenical tin is treated with a mixture of sodium hydroxide and sodium chloride to which small quantities of sodium nitrate are added from time to time. The slag, which contains the arsenic as sodium arsenate, is skimmed off the metal and boiled with water, and the clear solution is evaporated for the recovery of the arsenate. Eventually the mother-liquor is evaporated to dryness and the residue used again in the process. A. R. Powell.

Treating arsenic and antimony compounds and minerals. M. F. Coolbaugh and J. B. Read, Assrs. to Complex Ores Recoveries Co. (U.S.P. 1,597,018, 24.8.26. Appl., 4.3.22).—Arsenic and antimony ores containing sulphur are roasted in a slightly inclined, rotating, cylindrical furnace to convert the metals into arsenates, antimonates, and sulphates, which are subsequently extracted by a leaching process. A. R. Powell.

Casting iron in permanent molds at a regulated cooling rate. D. H. Meloche, Assr. to E. Holley (U.S.P. 1,597,861, 31.8.26. Appl., 3.3.23).—See E.P. 209,043; B., 1924, 751.

Electrochemical treatment of copper ores. H. S. MacKay (U.S.P. 1,598,296, 31.8.26. Appl., 19.5.25. Conv., 29.5.24).—See E.P. 238,962; B., 1925, 854.

Separating nickel and copper from copper-nickel mattes or other material. International Nickel Co., Assees. of R. C. Stanley (E.P. 240,789,

28.5.25. Conv., 30.9.24).—See U.S.P. 1,553,197; B., 1925, 888.

Electrothermic production of zinc. F. THARALDSEN (U.S.P. 1,598,176, 31.8.26. Appl., 25.5.22. Conv., 2.3.18).—See G.P. 395,105; B., 1924, 752.

Vanadium ores (U.S.P. 1,597,216).—See VII.

Enamelling metal (E.P. 254,452).—See VIII.

Tin (U.S.P. 1,598,295).—See XI.

Alkali metals (U.S.P. 1,597,231).—See XI.

XI.—ELECTROTECHNICS.

Refractory articles [for electric furnaces] from tungsten powder. J. HÄRDÉN (Chem. Met. Eng., 1926, **33**, 543—544).—Crucibles and cylindrical collars suitable for use as the heating medium in Northrup induction furnaces may be made by ramming a plastic mass of tungsten powder bonded with a 25% solution of dextrose into suitable steel moulds. After heating the moulded masses to 600° in a non-reducing atmosphere, they may be drilled, filed, or turned in a lathe. They are rendered hard and durable by heating at 1600° in a carbon tube resistance furnace, whereby the tungsten is converted into the carbide containing about 6% C. The fired articles have *d* about 12·2—12·8, whilst the electrical resistance varies from 21 to 43 microhms per c.c., according to the temperature and duration of firing. A temperature of over 2000° is readily obtained in the high-frequency induction furnace, and metals and alloys of high m.p. may be melted in magnesia crucibles placed inside the tungsten shells. A. R. POWELL.

See also A., Oct., 1013, **Electrolytic separations by graded potentials** (LASSIEUR).

PATENTS.

Preventing the formation of explosive gas mixtures or similar dangerous mixture in electrical apparatus immersed in oil. M. BUCHHOLZ (E.P. 241,228, 12.10.25. Conv., 11.10.24).—The formation of explosive gas mixtures etc. in electrical apparatus immersed in oil, *e.g.*, in oil switches, is prevented by causing a continuous and uninterrupted current of air drawn from the atmosphere to pass through the space above the level of the oil, whereby any fumes present are either discharged into the atmosphere or may be absorbed. J. S. G. THOMAS.

Electric furnace. C. E. TAYLOR (U.S.P. 1,596,902, 24.8.26. Appl., 20.11.20).—A complementary electrode is adjustable with regard to an electrode fixed at the bottom of a vertical casing. Material to be treated is fed from the top of the casing so as to surround the lower end of the adjustable electrode, and material fed to a point above the fixed electrode is confined to the region immediately surrounding the adjustable electrode. J. S. G. THOMAS.

Electric furnace for treating comminuted material. J. J. NAUGLE (U.S.P. 1,597,208, 24.8.26. Appl., 21.4.23. Renewed 7.11.25).—A substantially continuous cylindrical heating chamber has an inlet, an outlet, and a number of heating zones which can be separately regulated and controlled at different temperatures. Electrodes rotate within the chamber and carry means for forcing material through the chamber. J. S. G. THOMAS.

Electromagnetic induction heating. C. R. BURCH, N. R. DAVIS, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (E.P. 257,021, 20.5.25).—A material of low electrical resistivity is placed in a crucible of electrically conducting material which is subjected to an alternating electro-magnetic field of frequency f cycles per sec., such that the expression $\pi D\sqrt{2f/r} \times 10^9$ is greater than unity, D denoting the outside diameter of the crucible measured in cm., and r the resistivity of the material of the crucible in ohm-cm. Thereby eddy currents are induced in, and are substantially confined to, the crucible. J. S. G. THOMAS.

[Plates for] storage batteries. G. F. DOWNER (E.P. 257,019, 20.5.25).—Positive and negative lead plates of storage batteries are pasted with a substantially inactive paste of lead oxide and water, and then sulphated by soaking in a solution of sulphuric acid, loosening of the paste during sulphation being prevented by having the surface pores of the plates full of water on immersion in the acid. The plates are subsequently desulphated by electrical treatment in a small amount of substantially pure water, whereby the acid concentration of the electrolyte is raised approximately to normal strength. J. S. G. THOMAS.

Electrolytic production of alkali metals. P. E. HAYNES (U.S.P. 1,597,231, 24.8.26. Appl., 23.3.22).—A molten compound of the metal in a suitable bath is electrolysed, and the products are withdrawn separately as gas or vapour under less than atmospheric pressure. D. F. TWISS.

Electrodeposition of tin. H. R. MCILHENNEY, Assr. to VULCAN DETINNING CO. (U.S.P. 1,598,295, 31.8.26).—During the electrodeposition of tin the strength of the electrolyte is maintained by adding to it a tin compound that is insoluble in water but soluble in the products of the electrolysis as they are formed. A. R. POWELL.

Filaments for electron discharge devices. WESTERN ELECTRIC CO. From BELL TELEPHONE LABORATORIES, INC. (E.P. 257,125, 27.10.25).—A refractory metal, *e.g.*, nickel, is mixed with compounds of the alkaline earths, *e.g.*, barium and strontium carbonates, and heated to about 1200° in air or oxygen. The resulting product is crushed, mixed with a second refractory metal, *e.g.*, platinum powder, pressed, and heated to about 1600° *in vacuo*. The resulting product contains alkaline-earth oxides uniformly distributed throughout the platinum–nickel rod. J. S. G. THOMAS.

Decoration of electric incandescence lamps and bulbs. Z. HADNAGY and V. G. DE CHRISTIAN (E.P. 257,067, 16.7.25).—A diffusing coating for electric incandescence lamps consists of a cold varnish or enamel made of a solution of an alkali silicate, *e.g.*, sodium silicate, the concentration of which is 20—30%, according to the quantity and coarseness of grain of the pigment (if any) present in the coating. The addition of pigment, *e.g.*, finely powdered fuchsine, to the stronger solutions of silicate prevents the formation of bubbles and swellings in the coating. J. S. G. THOMAS.

Producing stannous chloride (U.S.P. 1,597,653).—See VII.

Production of burnish on iron or steel (E.P. 257,140).—See X.

XII.—FATS; OILS; WAXES.

Application of the counter-current principle to the steam deodorisation of saponifiable oils. W. BRASH (J.S.C.I., 1926, **45**, 331—333 T).—A description of the counter-current process for the deodorisation of oils is given. The theory of the process is discussed, and it is shown that towards the bottom of the deodorising tower a thinning out of the oil layers occurs, and that the cross-sectional area of the tower should diminish; the conditions necessary for the most economical use of the steam supply, and the variation of the height of the tower, with the thickness of the oil layers, the temperature and the speeds of efflux of the oil and steam, are derived. The advantages claimed over the unit deodoriser are economy of steam and short time of deodorising.

Oxidation of linseed and cottonseed oils. N. G. CHATTERJI and G. I. FINCH (J.S.C.I., 1926, **45**, 333—335 T).—The oxygen absorption of linseed and cottonseed oils has been ascertained by direct measurement of the volume of oxygen absorbed when air or oxygen was bubbled through the oil in a closed circulation apparatus (J.C.S., 1925, **127**, 2464) at 20°. No catalyst was used, and carbon dioxide and water were absorbed. The absorption of oxygen, after an induction period, reached a maximum identical with that calculated from the iodine value of the oils, assuming 2I = O.

Recovered fatty matter. C. DABOUST (Bull. Soc. d'Encour., 1926, **125**, 365—383).—The author discusses the various methods of fat recovery by boiling, autoclave treatment, acid treatment, and the action of solvents, particularly petrol. The treatment of bones for the extraction of bone grease, the recovery of skin grease in tannery operations, of grease from oily cleaning waste and from the refuse of slaughter houses, and of wool grease is described, with illustrations of washing machines, centrifuges, etc. A. RAYNER.

Unreliability of the Tortelli–Jaffé reaction [for marine-animal oils]. M. AUERBACH (Chem. Umschau, 1926, **33**, 163—164).—This reaction (cf. B., 1914, 1061), claimed to be characteristic of marine-animal oils before and after hydrogenation, is now found to be given by hardened vegetable oils and also by tallows from animals which have been fed on fish meal, whilst a definite reaction is not always obtained with marine-animal oils, the colour produced sometimes being violet. Hardened bean oil, from bean oil which did not give a positive result, gives a definite reaction, but in many other samples of hardened bean oil the result was negative. It is suggested that the chromogenic substance may be formed as a result of a particular method of hydrogenation. A. RAYNER.

Oils extracted from the head of a dolphin. H. MARCELET (Bull. Soc. chim., 1926, [iv], **39**, 1265—1275).—A more detailed account of work already published (B., 1926, 677).

Acetin or dichromate method [for glycerin analysis]. W. PRAGER (Z. deuts. Oel- u. Fett-Ind., 1926, **46**, 577—578).—An influential committee has been formed in Germany for the investigation of the relative efficiency of the two methods. At the formation of this committee it was stated that results by the acetin method were commonly 2% lower than with the dichromate method. Such statements are strongly repudiated by the author, on theoretical and practical grounds, and the relative advantages and possible accuracy of the two methods are discussed. A. RAYNER.

Free alkali in soap. W. ISMAILSKY (Z. Deuts. Oel- u. Fett-Ind., **46**, 545—546, 562—564).—The alcohol extraction method and the baryta method for determining free alkali in soap are untrustworthy, whilst the modification of the baryta method by Bosshard and Huggenberg yields satisfactory results only with soaps containing up to 10% of fatty acids, the errors being due to the absorption of alkali by the baryta soap, the difficulty of titration in presence of a bulky and sometimes coloured precipitate, and the presence of silicates. In an improved method 10 g. of soap are dissolved in 200 c.c. of freshly distilled water in a 400 c.c. flask, 20 c.c. of 30% barium chloride solution are added, the flask is loosely corked, and the contents are boiled and rotated until the precipitate agglomerates into a ball or a granular precipitate. The solution is cooled, the flask being corked meanwhile, and is filtered rapidly, and the precipitate washed with 100 c.c. of cold distilled water. The filtrate is titrated with $0 \cdot 1N$-acid, using phenolphthalein as indicator, and the result calculated to sodium hydroxide. W. G. CAREY.

Oxidation of drying oils. TARADOIRE.—See XIII.

Nutritive value of synthetic fats from fatty acids with an odd number of carbon atoms. OYAKI.—See XIX.

PATENTS.

Apparatus for extraction of oils, fats, waxes, resins, and greases from substances containing them. J. MACGREGOR, and E. SCOTT & Co., LTD. (E.P. 256,019, 25.6.25).—The material is treated with solvent in one or more percolators or extractors mounted above a steam-heated solvent recovery vessel with which the percolators are in communication by means of passages controlled by valves. When extraction has been carried to the desired extent the supply of solvent is cut off from the extractors, the solvent and oil are drawn off, and the treated material is caused to fall into the solvent recovery vessel, where it is subjected to the heat of the steam in the surrounding jacket or, where desired, subjected to open steam. The recovery vessel is vacuum-operated and when removal of solvent is completed, the treated contents are discharged. When continuous operation is desired the extracted material from the extractors, instead of going to the recovery vessel direct, is passed into a hopper, whence, by means of a worm-feed device consisting of externally heated tubes, the discharged material is ultimately delivered into a vacuum vessel, and then it passes through a discharge orifice controlled by a valve into a second vessel.

From this lowermost vessel the material can be discharged without breaking the vacuum in the remainder of the apparatus. H. M. LANGTON.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Rapid oxidation of drying oils; effect of antioxidising agents. F. TARADOIRE (Compt. rend., 1926, **183**, 507—508).—The spontaneous ignition of cotton waste soaked in a mixture of a drying oil, turpentine, and metallic drier is retarded or inhibited by antioxidising agents (cf. B., 1926, 135). Of a further series of organic compounds examined for this property, nitroso-derivatives of diphenylamine prevented ignition, whilst α- and β-naphthylamine, benzidine, pyridine, and quinoline retarded it for several hours. S. S. WOOLF.

Accelerated paint testing. M. SCHULZ (Farben-Ztg., 1926, **31**, 2879—2882).—From general considerations of atmospheric disintegrating influences on paints, the following scheme for rapidly reproducing the results of a lengthy period of weathering is arrived at. The paints under test are applied to iron plates and allowed to dry for at least three days, then "matured" for 24 hrs. at 80°. Subsequent treatment consists of six repetitions of the following cycle: hanging in distilled water at 20°, exposure to ultra-violet light while moist and again when dried, re-immersion in distilled water, enclosure in a damp atmosphere containing carbon dioxide, further exposure to ultra-violet light under damp conditions, then at 50°, enclosure in an atmosphere containing sulphur dioxide, exposure to damp at 35—40° with interpolated periods at —5°, and renewed action of ultra-violet rays. The results of some tests carried out by this method on zinc oxide–lithopone paints show agreement with a 2-year exposure to the atmosphere, in that the iron plates show increasing rusting with the increase in proportion of lithopone to zinc oxide in the protecting paint. The author stresses the value of these "physical paint tests" as a complement to chemical analysis. S. S. WOOLF.

Traffic paint. H. A. NELSON and S. WERTHAN (Ind. Eng. Chem., 1926, **18**, 965—970).—The composition and properties desirable for paints suitable for marking out directional lines on roads are studied by a series of tests on consistency, opacity, visibility by day (normal illumination) and night (oblique illumination), and resistance to combined weathering and abrasion. Most satisfactory results were obtained with lithopone (of non-greying quality), titanox, or zinc oxide as pigment, asbestine (talc) as inert filler (30% of total "pigment"), and a medium of high volatile content, the non-volatile portion consisting of tung oil–ester gum or tung oil–lime-hardened rosin, with or without the addition of linseed oil varnish. Appreciable quantities of lead pigments are not desirable owing to tendency to discoloration. White is the most suitable colour for this type of paint, as yellow and orange commercial traffic paints tested had lower visibility. S. S. WOOLF.

See also A., Oct., 993, **Phosphorescent sulphides of zinc** (GUNTZ).

PATENTS.

Manufacture of carbon black, unsaturated gases, and hydrogen. J. J. JAKOWSKY (U.S.P. 1,597,277, 24.8.26. Appl., 10.11.22).—The products of thermal dissociation of a hydrocarbon are passed through a cooling liquid. S. S. WOOLF.

Extracting turpentine, pine oil, and rosin [colophony]. E. P. STEVENSON, Assr. to A. D. LITTLE Co. INC. (U.S.P. 1,597,215, 24.8.26. Appl., 11.5.20).—Coniferous wood is treated with a coal tar hydrocarbon solvent of b.p. below that of turpentine. The extract is treated with hydrogen chloride to precipitate certain dark-coloured substances, and the solvent, turpentine, pine oil, and rosin are separated by distillation. S. S. WOOLF.

Obtaining phenol–aldehyde condensation products in a purified form. BAKELITE GES.M.B.H. (E.P. 246,833, 22.1.26. Conv., 27.1.25).—Phenol–aldehyde resins in alkaline solutions are precipitated by carbonic or other acid in the presence of hydrotropic salts, *e.g.*, soluble salts of aromatic or the higher fatty acids, especially sodium salicylate or alkali soaps. S. S. WOOLF.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Theory of needle-shaped rubber molecule. E. LINDMAYER (Gummi-Ztg., 1926, **40**, 2805—2807; cf. B., 1926, 714).—Unworked rubber has a formula $(C_5H_8)_{12}$ but when masticated undergoes fission into shorter $(C_5H_8)_6$ and $(C_5H_8)_3$ molecules; these shorter needle-shaped particles are in Brownian movement, and their rotation gives the optical effect of a round particle. The separation of "diffusion rubber" by dialysis from masticated rubber is explained by the presence of these molecules of various magnitude. At 75° the $(C_5H_8)_{12}$ molecule undergoes disaggregation into $(C_5H_8)_6$, the change being easily reversible, whereas the $(C_5H_8)_6$ and $(C_5H_8)_3$ molecules formed by mastication re-associate themselves much more slowly on account of their more drastic separation from one another. Calender grain is caused by orientation of the immobile $(C_5H_8)_{12}$ molecules; freezing of rubber at 14° is explained by the terminal coupling of two such molecules, the change being assisted by stretching. The gradual character of the softening on warming is due to the resins present, resin-poor rubbers showing a sharper change. Vulcanisation normally gives rise to $(C_5H_8)_6 \cdot S \cdot (C_5H_8)_6$ molecules, but overworked rubber needs a higher proportion of sulphur due to the formation of some $(C_5H_8)_3 \cdot S \cdot (C_5H_8)_3$ molecules; to vulcanite is ascribed the formula $(C_5H_8S)_6 \cdot S \cdot (C_5H_8S)_6$. The possibility of reclaiming rubber is dependant on the change $(C_5H_8)_6 \cdot S \cdot (C_5H_8)_6 \rightarrow (C_5H_8)_3 \cdot S \cdot (C_5H_8)_3 + 2(C_5H_8)_3$ with subsequent gradual polymerisation of the latter product to $(C_5H_8)_6$ and $(C_5H_8)_{12}$ molecules. D. F. TWISS.

Experiments with unmasticated rubber. W. C. DAVEY (J.S.C.I., 1926, **45**, 364—365 T).—The incorporation of varying amounts of glue produces effects in raw unmasticated rubber which are in some respects similar to those produced by vulcanisation. Comparative results of tests on vulcanised rubber prepared from unmasticated and masticated mixings are also given, showing the former to produce higher tensile strengths over a wider range.

Theory and practice of the acceleration of vulcanisation. F. EMDEN (Kautschuk, 1926, 1, 91—94, 110—112, 137—138, 158—161, 180).—A review of the theories as to the mode of action of accelerators, and of the present knowledge as to individual examples or groups of such agents, their activation by metallic oxides, their advantages and disadvantages. D. F. TWISS.

Antioxidants and their retarding action in the deterioration of rubber. L. E. WEBER (Ind. Eng. Chem., 1926, 18, 963—964).—Other factors than mere oxidation must be involved in the deterioration of vulcanised rubber, and further chemical investigation of this deterioration is needed. Some organic accelerators exert an antioxidant effect; certain condensation products of aldehydes with an aromatic amine, *e.g.*, of aldol and α-naphthylamine, are very feeble accelerators of vulcanisation, but have a marked preservative action on vulcanised rubber. It is possible that these antioxidants do not act catalytically, but protect the rubber by undergoing preferential oxidation themselves. D. F. TWISS.

See also A., Oct., 1003, **Consistency of solutions of rubber in benzene** (HERSCHEL and BULKLEY).

XV.—LEATHER; GLUE.

Chrome tanning at the isoelectric point of collagen. R. M. COBB and F. S. HUNT (J. Amer. Leather Chem. Assoc., 1926, 21, 454—464).—Four series of chrome tanning liquors were prepared containing 1% of salt, 1·5% Cr_2O_3 and sodium formate or acetate, or 0·75% Cr_2O_3 and sodium formate or acetate respectively. The basicity was 33%. The p_H value was adjusted by adding acid or alkali, and pieces of pelt were tanned in each series at different p_H values for a definite period, after which the chromium content was determined. It increased as the p_H value increased. There was no break at the isoelectric point, but a steady unbroken increase from p_H 3·0 to 6·0, hence chrome tanning cannot be a chemical reaction between free ions, but the chromium unites with the collagen by means of secondary valencies. The degree to which pelt absorbs acid from the pickle must greatly affect the extent of tannage. D. WOODROFFE.

South Indian tanning materials. K. S. CHOUDARY and E. YOGANANDAM (J. Soc. Leather Trades Chem., 1926, 10, 222—228).—The analyses of eight common South Indian tanning materials are: konnan (*Cassia fistula*) tans 11·2%, non-tans 12·1%; avaram (*C. auriculata*), tans 16·8%, non-tans 10·1%; wattle (*Acacia decurrens*), tans 35·8%, non-tans 14·4%; divi divi (*Caesalpina coriaria*), tans 33·6%, non-tans 26·4%; sumach (*Anogasis latifolia*), tans 13·0%, non-tans 9·1%; babool (*Acacia arabica*), tans 7·6%, non-tans 4·6%; gothar (*Zizyphus xylopra*), tans 12·8%, non-tans 17·7%; myrobalans (*Terminalia chebula*), tans 27·1%, non-tans 10·5%. Gothar and myrobalans contain 19·2% and 16·8% of sugar respectively, calculated on the tannin. Myrobalans and wattle have the most favourable proportion of tans to non-tans. Divi divi, myrobalans, and sumach lost most tannin, and wattle and avaram the least, when infusions were kept. The p_H value of all the liquors except those of divi divi (3·5) and myrobalans (3·4) was near 4·7. Divi divi, myrobalans, sumach, and babool are the liquors in which the non-tannins diffuse more rapidly. The materials that lose most on keeping diffuse most freely. The colour of the liquor has been shown to depend on whether the material has been extracted hot or cold. Avaram bark yields a less reddish coloured infusion when extracted hot, whereas divi divi gives a more reddish colour when extracted hot. D. WOODROFFE.

[Ultrafiltration of vegetable tanning solutions.] R. J. BROWNE (J. Soc. Leather Trades Chem., 1926, 10, 235—237).—A reply to criticisms by Thomas and Kelly (B., 1926, 290). The author's ultrafiltration experiments (B., 1923, 989A) were purely mechanical and there was no chemical action. Thomas and Kelly did not test the permeability of their membranes, and did not use high pressures in every case. The author prepared various types of collodion membranes, all of which retained tannin if their impermeability was suitable. D. WOODROFFE.

Action of sodium sulphate in synthetic tanning materials. E. WOLESENSKY (U.S. Bur. Standards, Tech. Papers, 1926, 20, 529—544).—Wet chromed hide powder removes sulphuric acid from weak solutions, and from a 0·5% solution of sodium sulphate acidified with 0·5% of acetic acid. Prolonged washing did not remove the sulphuric acid. Experiments showed that sulphuric acid in combination with hide substance cannot be completely displaced by a synthetic tannin whereas combined synthetic tannin can be removed from hide substance by treatment with sulphuric acid, or with a mixture of sodium sulphate and acetic acid solutions. It follows that if a hide is treated with a synthetic tannin containing a soluble sulphate, both sulphuric acid and synthetic tannin will combine with the hide. The neutralisation of the excess sulphuric acid in the manufacture of synthetic tannins by means of sodium hydroxide or any other reagent which yields a soluble sulphate, does not prevent the sulphuric acid from reacting with the hide during the tanning process. It is possible that the sulphuric acid combined with the hide is not injurious to the latter, but this remains to be determined. The behaviour of sodium sulphate in synthetic tannins must cause an appreciable error in the non-tans determination unless the free sulphuric acid and soluble sulphates are precipitated by means of an equivalent amount of barium acetate. The synthetic tannins used included phenolsulphonic and cresolsulphonic acids condensed with formaldehyde. D. WOODROFFE.

Fermentation of divi divi liquor. II. Acidity of divi divi liquor. K. S. CHOUDARY and E. YOGANANDAM (J. Soc. Leather Trades Chem., 1926, 10, 237—239; cf. B., 1925, 218, 412).—Divi divi liquors (*d* 1·015) extracted in the hot and in the cold were kept and analysed periodically for total acidity and p_H value. The total acidity increased to a maximum about the 9th day, then diminished and rose again to a maximum on the 74th day, the maximum on the latter day being 30% more. The p_H value was 3·7, and changed very little. It was 3·3 at the end of the period. The total

acidity was greater in the liquors in which the pods were allowed to remain. D. WOODROFFE.

Direct measurement of plumping power of tan liquors. [Report of committee of the American Leather Chemists' Association.] R. E. PORTER (J. Amer. Leather Chem. Assoc., 1926, **21**, 425—435; cf. B., 1925, 603).—Dry sifted hide powder (1·5 g.) is weighed into each of two cylinders 230 mm. long and 26 mm. inside diam., covered with 50 c.c. of water, and kept for 3 hrs. with 5 stirrings. Measuring plungers made from the bulb and upper stem of a 15 c.c. pipette and fitted at the lower end with a perforated brass disc, are allowed to drop slowly on the hide powder in the cylinders. After 5 min. the height of the hide powder is read on the scale to the nearest mm. The plungers are removed and the excess soak water siphoned off to a line giving a capacity of 26·66 c.c. The compressed hide powder is well stirred up in the 25 c.c. of water, 75 c.c. of the tan liquor are pipetted into the cylinder, which is kept for 24 hrs. with occasional stirring, then the plunger is introduced as before and the new reading taken. The final reading divided by the initial reading gives the plumping ratio of the tan liquor. This method shows higher sensitivity and involves the use of hide powder and simple apparatus, The calf pieces in the Wilson and Gallun method are difficult to obtain uniform. D. WOODROFFE.

Determination of moisture in leather. [Report of a committee of the American Leather Chemists' Association.] F. P. VEITCH and T. D. JARRELL (J. Amer. Leather Chem. Assoc., 1926, **21**, 435—445; cf. B., 1925, 683).—It is recommended that the toluene distillation method, Bidwell–Sterling modification (cf. B., 1926, 839), be made official for the determination of moisture in leather. D. WOODROFFE.

Determination of fat in leather. D. WOODROFFE (J. Soc. Leather Trades Chem., 1926, **10**, 219—221).—Samples of chrome and vegetable tanned leathers, fat-liquored either with cod oil or with degras fat liquors, have each been extracted with light petroleum in three ways, from the air-dry sample, from the dried leather, and from the leather which had been dried at 105° and then exposed to a humid atmosphere. The extract from the dried leathers was less than that from the air-dry sample and the difference could not be accounted for by the extraction of water-soluble matter (cf. Hey, B., 1923, 65 A). There was no evidence to show that oxidation of the oils took place when the above leathers were dried by heating at 105°. The greater amount of extract from air-dry leather is probably due to moisture. D. WOODROFFE.

PATENT.

Finishing and colouring of leather. M. C. LAMB, and P. SPENCE & SONS, LTD (E.P. 254,350, 31.12.24).—In finishing and colouring leather a previously prepared paint or dope is applied, made by adding to titanium tannate natural or artificial organic dyestuffs or mineral pigments. The dope may be mixed with mucilage prepared from gums, Irish moss, linseed, etc., and applied by brushing or spraying. H. MOORE.

XVI.—AGRICULTURE.

Chemical methods for determining whether soils need nitrogenous or potassium fertilisers. A. NEMEC (Compt. rend., 1926, **183**, 483—485; cf. B., 1926, 763).—Nitrates are determined in a water extract of soil (100 c.c. water to 30 g. soil for 1 hr.) after allowing nitrification to proceed in the moist soil for 14 days. Comparison of the figures obtained with the results of field experiments with sodium nitrate indicate that, for sugar beet, soils containing less than 25—30 mg. of nitric nitrogen per kg. of air-dry soil may be expected to respond to nitrogenous fertilisers. Corresponding figures for barley are 22—28 mg. and for oats, 20 mg. of nitrogen. Potassium is determined in a water extract, cleared by adding calcium carbonate and heating on the water-bath, by Cameron and Failyer's method (cf. B., 1903, 1259). The figures above which potassium fertilisers are not likely to be effective are, for sugar beet 37 mg. K_2O, for barley 10—20 mg., for oats 13—19 mg., for potatoes about 25 mg. per kg. of air-dry soil. C. T. GIMINGHAM.

Influence of the application of superphosphate and sodium nitrate on the chemical composition of the stem and leaf of pasture cuts of cocksfoot. T. W. FAGAN and R. E. EVANS (Welsh J. Agric., 1926, **2**, 113—116).—The ratio of true to crude protein was higher in the leaves, which were richer in these substances and in ash than the stems. The stems contained more iron and chlorine, and the leaves more calcium. Application of superphosphate increased the fibre and decreased the chlorine content; sodium nitrate decreased the phosphate and calcium content but increased the protein content. A. A. ELDRIDGE.

Insecticidal value of certain war chemicals as tested on the tent caterpillar. F. G. BRINLEY (J. Agric. Res., 1926, **33**, 177—182).—A number of compounds used during the war (chiefly organic arsenic compounds and metal stearates and resinates) have been tested, both as stomach poisons and contact insecticides, on eastern tent caterpillars (*Malacosoma americana*). Most of the compounds that proved toxic to the insects were also so toxic to plants as to have no practical value. Iminodiphenylenearsenious oxide was an exception. It was equal in toxicity to lead arsenate and was not injurious to bean plants. It also showed promise as a contact insecticide. The compound is, however, highly irritant, and great caution is required in handling it. Copper stearate and resinate were definitely repellent to the insects, though comparatively non-toxic; they have good spreading and adhering qualities. C. T. GIMINGHAM.

See also A., Oct., 1018, **Electrometric titration of phosphates** (BODFORSS).

PATENTS.

Preparation of calcium nitrate capable of being strewn. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 242,990, 11.11.25. Conv., 11.11.24).—Substantially anhydrous calcium nitrate, containing up to 17% of nitrogen, is mixed with a small proportion (about 0·1%) of solid hydrate of calcium nitrate at a temperature below the m.p. of the hydrate. This removes the

tendency of anhydrous calcium nitrate to effloresce, and maintains the product in a condition fit for distribution in the field for a considerable time. C. T. Gimingham.

Manufacture of fungicidal and insecticidal media. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 257,171, 19.2.26).—Copper sulphate is heated with an alkaline-earth carbonate in the presence of water. For example, 40 kg. of ground anhydrous copper sulphate are mixed with 60 kg. of dry finely divided chalk; 20 litres of water are stirred in to form a paste, which is heated to about 100°. The mass is dried and powdered; other insecticides or fungicides may be added. The powder thus prepared is used for dusting and is distinguished by great adhesiveness. C. T. Gimingham.

XVII.—SUGARS; STARCHES; GUMS.

Precipitation of calcium phosphate under various conditions, with special reference to sugar factories. R. G. W. Farnell (J.S.C.I., 1926, **45**, 343—347 T).—The formation and precipitation of calcium phosphate was studied in aqueous solution, in sucrose solution, and in a solution containing cane colloids. The methods employed were similar to those described in the precipitation of calcium sulphite (cf. B., 1926, 72), and the following factors were varied: (1) time of precipitation (5—60 min.); (2) temperature of precipitation (20—100°); (3) extent of neutralisation of phosphoric acid (p_H 2·2—10·6); (4) initial concentration of phosphoric acid (0·0070—0·0934 g. P_2O_5 per 100 c.c.). The experimental results are represented graphically. Whilst sucrose had no effect in delaying the precipitation of tricalcium phosphate or altering the nature of the precipitate, the precipitation was much affected by the salts and colloids present in cane syrups and sugars. The greater the amount of P_2O_5 naturally occuring in the juice, the greater is the precipitation of calcium phosphate on adding lime, and the more efficient is the clarification. If the clarified juice, after liming, heating, and settling for 1 hr., leaves the clarifiers at p_H 7·0 practically all the phosphate will have been precipitated. Phosphoric acid and phosphate generally constitute the principal buffer substances of the cane juice. The drop in p_H noticed in sugar factories on heating a limed juice appears to be partly due to the precipitation of calcium phosphate and its subsequent hydrolysis to free phosphoric acid and the more basic hydroxyapatite, which is the only phosphate of lime which is not decomposed by water.

Sucrose as an acid. [New theory of the formation of molasses.] J. Dedek and P. Terechov (Z. Zuckerind. Czechoslov., 1926, **50**, 349—355).—Hydrogen-ion concentration determinations and mol. wt. measurements lead the authors to annunciate a theory of the formation of molasses based on the supposition that sucrose plays the part of a monobasic acid in dilute solution, the valency rising in higher concentrations, and being dependent on the ratio of sucrose to alkali. In general, the stronger the concentration of the salts present, and the weaker their acids, then the greater the amount of sucrose entering into the reactions. In the case of any molasses the molar concentration of the sucrose practically agrees with the equivalent concentration of the potassium and sodium ions. J. P. Ogilvie.

Surface tension of sugar factory juices. V. Sázavský (Z. Zuckerind. Czechoslov., 1926, **50**, 378—380, 423—426).—Using Traube's stalagmometer (cf. B., 1922, 121 A), the surface tension of saccharine solutions can be determined with sufficient accuracy provided the density is not more than 30° Brix. Above that degree the results are less certain, and such solutions require to be diluted and kept for a time. Lindfors' results obtained by using the Du Noüy apparatus (B., 1926, 72) are not considered accurate within 10%. Surface tension determinations depend largely on the p_H of the solution examined, and addition of alkali causes a marked alteration of the result. J. P. Ogilvie.

Practical methods of p_H measurement for the control of cane juice defecation. R. T. Balch and H. S. Paine (Intern. Sugar J., 1926, 28, 425—429).—Apparatus consisting of a recording potentiometer, a saturated potassium chloride–calomel electrode, a salt bridge, a continuous-flow electrode chamber, a tungsten or similar type of electrode, and a small centrifugal pump has been adapted for the control both of intermittent and of continuous liming. At present in continuous liming the recording potentiometer may be made to record from one to a number of circuits simultaneously, to operate such signals as warning lights or bells when the reaction does not fall within the desired limits, and, in proportion to the change in reading, to operate an electro-magnetic valve controlling the rate of lime addition. J. P. Ogilvie.

Simple method of measuring hydrogen-ion concentration and its significance in the beet-sugar industry. F. Tödt (Z. Ver. deut. Zuckerind., 1926, 494—508).—A colorimetric spotting plate procedure is recommended, comparisons being made with a colour chart (printed in the original). Indicators are used in the form of 0·04% alcoholic solutions, and molasses is diluted to 10° Brix and other products to 20—40° Brix. Test-papers are recommended only for approximate results; any universal indicator containing bromothymol-blue as the "alkaline" constituent is condemned so far as sugar factory products are concerned. First carbonatation juice contains strong bases and salts of very weak acids and very weak bases; in the second carbonatation the strong bases have for the greater part been eliminated, whilst in the thick-juice the very weak bases are no longer to be found, having disappeared during the evaporation. Juice from damaged roots shows a low p_H value, as it contains stronger acids and weaker bases, as compared with normal juice. (Cf. B., 1925, 416.) J. P. Ogilvie.

Applications of [electrical] ash analysis in [sugar] factory control. H. Lundén (Z. Ver. deut. Zuckerind., 1926, 510—516).—Yield formulæ which are based on values for the mineral matter of sugar factory and refinery products are more accurate and simpler, and are rapidly calculated when the ash is determined by the electrical conductivity method (B., 1925, 1003). This method enables the quality of

white sugars to be gauged according to their ash content, the finest refined having a value of 0·002, average refined, 0·002 to 0·003; and "melis," 0·025%. The amount of ash in a sugar is normally $\frac{1}{80}$th of that of the massecuite from which it is crystallised, and the colour about $\frac{1}{25}$th. In the case of persistent impurities this effect may be only $\frac{1}{6}$th or even only $\frac{1}{2}$.

J. P. Ogilvie.

Defects of Fehling's solution. R. Ofner (Z. Zuckerind. Czechoslov., 1926, **50**, 355—356, 360—364, 370—372).—Fehling's solution as a reagent for the determination of invert sugar even in first-product raw beet sugars is insufficiently reliable for the following reasons: considerable oxidation of an inconstant degree takes place in the presence of sucrose; it is too sensitive, results depending on deviations of procedure which are not realised; reduction depends too much on the regularity of ebullition; there is great inclination to delay in boiling; and certain impurities have an important influence on the separation of cuprous oxide.

J. P. Ogilvie.

Patents.

Extraction of sugar from sugar cane. F. Kessler (E.P. 250,889, 12.6.25. Conv., 18.4.25).—The crushed raw cane is fed on to a sliding plate, where it is sprayed with the solvent, and then slightly compressed by means of a spring-journalled compressing roller. It is also treated from above or below with compressed air or steam from a blowing box, and finally passes to the extracting pressing rollers for the removal of the concentrated extract.

F. R. Ennos.

Producing a reaction of lime and sucrose in the manufacture of sugar from beets. R. W. Shafor, E. Morrison, R. J. Brown, L. A. Stenger, and A. R. Nees, Assrs. to Great Western Sugar Co. (U.S.P. 1,593,782, 27.7.26. Appl., 15.8.24).—The apparatus comprises a bank of horizontal flues, interconnected for the passage of a cooling liquid along a zig-zag course, the flues extending transversely across a space constituting the circulation chamber, in which the reaction between the lime and sucrose is accomplished. The chamber is divided by vertical partitions to provide a zig-zag path for the material, whilst the outer compartments of the circulatory chamber connect at their lower ends with circumferential egress ports of rotary centrifugal pump impellers in order to promote movement of the liquid.

J. P. Ogilvie.

Processes for obtaining tricalcium saccharate. C. Steffen, jun. (E.P. 255,175, 22.7.26).—Molasses or sugar solution is subjected on the way to the main collecting receiver immediately after the addition of calcium oxide (in powder) to a vigorous mixing, after which the liquid is passed on to the main collecting receiver and repeatedly circulated through the whole apparatus until the desired amount of tricalcium saccharate has been formed. This procedure results in economy in the use of lime. J. P. Ogilvie.

Apparatus for evaporating and crystallising sugar and other solutions. F. Lafeuille (E.P. 252,686, 15.4.26. Conv., 20.5.25).—A cylindrical vessel capable of rotation about its axis and provided with a set of circulation tubes, has a vacuum chamber of larger diameter as an extension. By the passage of steam or cold water through the tubes, the apparatus may be applied alternatively as an evaporator, under the vacuum, or as a crystalliser. F. R. Ennos.

Molasses-product and method of producing the same. S. D. Wilkins, H. C. Reiner, and E. C. Gould (E.P. 257,691, 15.6.25).—Molasses is heated to about 90° to lower its viscosity, mixed with a suitable colloid, *e.g.*, casein or skimmed milk solids, and evaporated to dryness in a dehydrating apparatus. The resulting product is a non-hygroscopic, fluffy powder which, while it may be applied to substantially all the present-day uses of liquid molasses, is more readily and economically handled. F. R. Ennos.

XVIII.—FERMENTATION INDUSTRIES.

Resinification of the α-bitter acid of hops (humulone) by molecular oxygen. W. Windisch, P. Kolbach, and J. Yofé (Woch. Brau., 1926, **43**, 349—353, 359—363, 369—372, 379—383).—During the boiling of wort with hops the humulone, which is the most valuable of the bitter principles of hops, is partially oxidised and converted into resinous substances. Similar changes occur during the storage of hops, and cause deterioration in their brewing value. The humulone is transformed first into α-soft resins (soluble in light petroleum), and finally into insoluble hard resins. For these changes both oxygen and water are necessary. The first step is peroxide formation at a double linking, and by uptake of water the linking is broken and an acid is formed as one of the products. In the pure dry state, humulone is very stable. Exposed to the atmosphere it is oxidised to the extent of 30—40% in 2 months at the ordinary temperature and in a few hours when heated near its m.p. It is very stable when dissolved in strong alcohol, but on addition of water it becomes very readily oxidisable as the limit of solubility is approached. Considerable oxidation occurs also during the evaporation of solutions in light petroleum, and for this reason, in the analysis of hops, light petroleum extracts which have been evaporated to dryness to determine their content of dry substance should not afterwards be used for the determination of humulone. In presence of certain readily oxidisable substances humulone oxidises much more rapidly than when pure; amongst such accelerating agents are hop oil and certain soft resins formed by the oxidation of humulone itself. On the other hand, the hard resin formed from humulone appears to protect it from oxidation, for under most conditions humulone which has undergone oxidation to the extent of 30—40% oxidises further at a very much slower rate. Heating fresh hops probably tends to preserve the humulone during subsequent storage, by driving off volatile matters, such as hop oil, which would accelerate its oxidation, and by melting the humulone and thereby reducing its surface. Heating of hops which have already been stored may be harmful, since substances present which have already taken up oxygen and formed peroxides, may transfer it to the humulone. Siller's method for determining humulone by precipitation with lead acetate

needs further study, for lupulin and hops often contain hard resins which may be precipitated under the same conditions. J. H. Lane.

Determination of the colour of malt. V. Bermann (Woch. Brau., 1926, **43**, 309—312).—Adler has constructed an apparatus for measuring the colour of malt extracts in accordance with the method previously described (B., 1925, 820; 1926, 170). In terms of Ostwald's theory of colour, the apparatus actually measures the "white-content" of the colour of the malt extract, and this by difference from unity gives the real colour-content ("Vollfarbe"), since for moderately clear liquids the "black-content" is negligibly small. J. H. Lane.

Colorimeters based on Ostwald's theory. F. Mestan (Woch. Brau., 1926, **43**, 312—316).—From a comparison of Adler's apparatus (see preceding abstract) with Hahn's instrument and Ostwald's chrometer, the author concludes that the first is likely to prove most suitable for the examination of malt extracts, but it could be improved in several important respects. J. H. Lane.

Development and nutrition of yeast. III. A. Tait and L. Fletcher (J. Inst. Brew., 1926, **32**, 385—414; cf. B., 1922, 724 a; 1923, 792 a).—Yeast kept for a day or two in its own beer after complete fermentation and then seeded into fresh wort exhibits a lag-phase in growth corresponding to a building-up process during which there is an appreciable assimilation of nitrogen. The influence of temperature, during storage of the yeast, in increasing the lag-phase points to the endocellular changes being of an enzymic nature, the velocity of the changes being affected by the hydrogen-ion concentration of the storage liquid. Storage of yeast in a nitrogen-free carbohydrate solution lessens the lag period. There is a marked difference between unwashed and water-washed yeast during storage, the washed yeast giving a greatly lessened lag-phase and a smaller proportion of dead cells. The washed and unwashed yeasts differ in their behaviour when stored in air, carbon dioxide, and nitrogen. With one exception, *i.e.*, the acid content of yeast stored in carbon dioxide, the amounts of acid, "total" nitrogen, and "formol" nitrogen are greater in the unwashed yeast. The washing almost entirely removes the interstitial and internal alcohol, the presence *per se* of which, however, has no effect upon the changes taking place during storage. Substances adsorbed on the surface of the yeast, and having a clogging effect, may be partially removed by washing, while the internal hydrogen-ion concentration of the cells is hardly, if at all, affected. Washing brings about an alteration in the course of autofermentation. Pressed unwashed yeast cannot get rid of its poisonous by-products, suffers from toxic substances, and quickly dies, this being followed by rapid liquefactive autolysis. Placed in fresh wort the unwashed yeast cells have first to expel the accumulated poisonous by-products and then, or concurrently, to absorb the carbohydrate and nitrogenous matters necessary to rebuild the starved plasma; in the case of the washed variety, although by-products and toxicity may be less in amount, still the yeast has also to rebuild its depleted plasma. Accordingly, the length of the lag-phase period must be some function of the extent of the cell disintegration. C. Ranken.

Colour changes of beer during the primary fermentation. W. Wanderscheck (Woch. Brau., 1926, **43**, 391—395, 403—408).—As a rule brewery worts become appreciably lighter in colour during fermentation. In the production of very pale bottom-fermentation beers of the Pilsen type it was found that in some cases the fermented beers were slightly darker than the original worts. A study of these cases led to the following conclusions:—The colour of wort fermented by freshly propagated pure-culture yeast diminishes immediately after pitching and throughout the fermentation. The yeast takes from the wort certain colouring matters, possibly protein-tannin compounds, which accumulate on it during successive fermentations until they begin to redissolve. In the cases studied this occurred after 6—7 fermentations. The re-solution of the colouring matters is inhibited by air but favoured by carbon dioxide. Hence, yeast which has been repeatedly used may extract colour from wort at the commencement of fermentation and yield colouring matters to the wort during the later stages. This is much more likely to occur with bottom-fermentation than with top-fermentation, owing to the much greater duration of the former. Washing the yeast with water and sifting, between successive fermentations, only partially removes the colouring matters in question, but treatment with dilute sodium carbonate solutions is much more effective. Darkening of wort during fermentation is not caused by the use of discoloured hops, nor by flocculent matters precipitated during wort boiling; if the latter remain in the beer during fermentation they tend to decolorise rather than to colour the wort. The main factors which affect the phenomenon are the character of the yeast and particularly the method employed for cleansing it, and the character of the malt used. The changes in the colour of wort during fermentation do not correspond with the changes in the concentration of hydrogen ions. J. H. Lane.

See also A., Oct., 1061, **Action of oxygen on alcoholic fermentation of sugar** (Gorr and Perlmann); **Action of ammonium salts on yeast fermentation** (Zeller); **Fermentation of sugars etc. by yeast juice and fresh yeast** (Neuberg and Kobel).

Patents.

Process for making fresh yeast lasting. L. J. J. Lindemann, Assr. to T. P. Hodge (U.S.P. 1,596,279, 17.8.26. Appl., 15.9.24).—Finely-divided yeast is suspended in lime or other alkaline water at 33—43° with or without the addition of a small quantity of sugar, and the whole gently agitated until tests show that the glycogen has been removed. F. R. Ennos.

Refining alcohol. E. A. Barbet (U.S.P. 1,598,548, 31.8.26. Appl., 14.11.22. Conv., 15.11.21).—See E.P. 189,136; B., 1923, 1193 a.

XIX.—FOODS.

The miscometer: an apparatus for obtaining composite samples [*e.g.*, of milk]. J. Houston (Analyst, 1926, **51**, 453—455).—Miscible liquids, *e.g.*, milk samples, may be quickly and accurately mixed, and a definite quantity delivered, without the use of pipettes, by using an apparatus consisting essentially of two cylinders, one graduated for measuring and the other for mixing, and both fixed to a stand. They are connected at their upper ends through a four-way stopcock (which in one position connects the measuring chamber and suction pump and at the same time allows air to enter the mixing cylinder, while when turned through 90° the connexions are reversed) to a device for creating partial vacuum, and at their lower ends by a glass tube. At the lower end of the mixing chamber is a stopcock so made that when closed a measured quantity (11 c.c.) flows into the narrower end, and when opened into a butyrometer, and at the same time the residue flows out through the side tube. By suitable manipulation the samples are successively drawn into the measuring chamber, the mixture is transferred to the mixing chamber, and air bubbled through. The 11 c.c. and residue of the composite sample leave the stopcock at the same time. D. G. Hewer.

Nutritive value of synthetic fats containing fatty acids of an odd number of carbon atoms. J. Ozaki (Proc. Imp. Acad. Tokyo, 1926, **2**, 12—14).—Feeding experiments with rats, in which the fat was supplied in the form of the pure glycerides of fatty acids with odd numbers of carbon atoms up to 17, prove that such fats have a nutritive value equal to or greater than that of natural fats and in general that this value tends to diminish as the acid involved increases in mol. wt. A definitely noxious effect was, however, observed with propionin (glyceryl tripropionate), undecylein, and *iso*valerin, the last being particularly marked in its effects. Saturation of undecylein to undecylin rendered it nutritious. G. M. Bennett.

Composition and maturity of maize. T. H. Hopper (N. Dakota Agric. Exp. Sta. Bull., 192, 1925).—The percentages of the following constituents increased with maturity—ear: dry matter, ether extract, nitrogen-free extract, also ash, protein, and fibre in the dry matter; stover: dry matter, also ash, and fibre in the dry matter; fodder: dry matter, ether extract, nitrogen-free extract. In the fodder the ash, protein, and fibre decreased, whilst in the stover the ash, protein, and nitrogen-free extract in the dry matter remained unchanged. The chemical evidence indicates that the maize plant is mature at the glazed stage. A. A. Eldridge.

Determination of acid-hydrolysable carbohydrates in green plant tissue. V. H. Morris and F. A. Welton (J. Agric. Res., 1926, **33**, 195—199).—With samples of green plant tissue preserved in alcohol, it is unnecessary to clear, or even neutralise, the hydrolysed solution before determining the reducing power. The only disadvantage of omitting clearing is that the precipitate of cuprous oxide is liable to be very finely divided; the use of a centrifuge is recommended. C. T. Gimingham.

See also A., Oct., 1065, **Preservation of vitamin-C in dried orange juice** (Humphrey).

Pectin. Nanji and Norman.—See XX.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Pectin: Micro-method for the determination of methyl alcohol, and its application to the study of the conditions governing the de-esterification of pectinogen. D. R. Nanji and A. G. Norman (J.C.S.I., 1926, **45**, 337—340 T).—Methyl alcohol in dilute solution is oxidised completely to carbon dioxide and water by boiling with alkaline permanganate solution for 3 min. under reflux. Oxalic acid is added, followed by sulphuric acid while hot, and the mixture titrated back with $N/20$-permanganate solution. The alcohol can be removed quantitatively from dilute solution by a single distillation, with certain precautions. As other volatile substances are absent from pectinogen and de-esterified pectinogen solutions, the alcohol can be determined directly in the distillates. The total methoxyl content of pectinogen is determined by treating it overnight in a closed vessel with $N/20$-sodium hydroxide solution, adding sulphuric acid to give p_H 4·5, and determining the alcohol directly in the distillate. In considering the results, it is essential to determine the yield of calcium pectate from pectinogen, as unesterified uronic acid complexes are often present to the extent of as much as 30%. The methoxyl contents hitherto recorded have been uniformly too low, as the presence of such impurities has been overlooked. The methoxyl content of a pure pectinogen preparation (11·76%) is found to agree closely with that required for a tetramethyl ester of pectic acid. Tri-, di-, and mono-methyl esters exist having methoxyl contents of 8·94, 6·04, and 3·06% respectively. The degree of de-esterification of the pectinogen molecule is shown to be the most important factor in jellying phenomena, satisfactory jellies being only given by the tetra- and tri-methyl esters. All those conditions likely to produce de-esterification above a certain degree must therefore be avoided during the manufacture of pectin substances, jams, etc. Results are given showing the degree of de-esterification produced under various conditions.

Treatment of sheep-rot virus with aldehydes. E. Ducloux and G. Cordier (Compt. rend., 1926, **183**, 486—488).—Vaccines prepared by attenuation of the virus of sheep-rot by addition of fatty aldehydes confer immunity on treated sheep and are safe in use. Preparations from rot pustules, to which formaldehyde was added at the rate of 2·5 pts. per 1000 or less, when injected subcutaneously (1 c.c. per animal) caused little or no reaction and protected against subsequent injection, 15 or 16 days later, of virulent lymph. Similar results were obtained with acetaldehyde and butaldehyde. The vaccines so prepared kept well. C. T. Gimingham.

Essential oils of Sardinian aromatic plants. III. Essence of *Thymus herba barona* and of *Thymus capitatus*. E. Puxeddu (Annali Chim. Appl.,

1926, **16**, 323—332; cf. B., 1925, 690).—The following data have been determined, the first values being those of the original oil, the values in brackets those of the oil re-distilled in steam. *Thymus herba barona* gives 1—1·36% of oil, red (yellow), d^{18} 0·9304 (0·9232), $[\alpha]_D^{18}$ (—4·83°), n_D^{20} 1·4925 (1·4970), solubility in 80% alcohol 1 : 1·5 (1 : 1·4), acid value 7·35 (9·90), ester value 3·80 (6·00), saponification value 11·22 (15·90), after acetylation 183·46 (187·26); phenol content 60% (mainly carvacrol). *Thymus capitatus* gives 0·3—0·6 of oil, d^{13} 0·9385, $[\alpha]_D^{13} - 1·6°$, n_D^{13} 1·5030, solubility in 80% alcohol 1 : 2, acid value 10·57, ester value 1·28, phenols 66%, these values referring to the oil after distillation in steam. E. W. Wignall.

See also A., Oct., 1012, **Catalytic oxidation of methane** (Medveev). 1042, **Identity of uncineol with eudesmol** (Penfold). 1049, **Determination of methoxyl** (Nierenstein). 1062, **Isolation of insulin** (Funk). 1063, **Crystalline insulin** (Abel). 1064, **Ovarian hormone** (Ralls and others, also Hartmann and Isler.).

Aktivin in analysis. Jangmichl and Hackl.—See VI.

Patents.

Process of combining ethylene with sulphuric acid. J. N. Compton, Assr. to Carbide and Carbon Chemicals Corp. (U.S.P. 1,598,560, 31.8.26. Appl., 29.5.23).—Ethylene is absorbed in a bath containing 20—90 mols. of ethylene per 100 mols. of sulphur trioxide. The composition of the bath is maintained by regulated additions of sulphuric acid. B. Fullman.

Dehydrating formic acid. Chem. Fabr. auf Actien (vorm. E. Schering) (E.P. 250,199, 12.3.26. Conv., 6.4.25).—The acid is treated with an anhydrous compound capable of retaining water of crystallisation with great tenacity, in sufficient quantity to form a stable hydrate with the water present in the acid, *e.g.*, with sufficient anhydrous copper sulphate or magnesium sulphate to yield a salt containing 1 mol. of water of crystallisation, after which the acid is recovered by distillation at the ordinary pressure or *in vacuo*, after filtering off the hydrated compound, if necessary. L. A. Coles.

Making halohydrins. H. Essex and A. L. Ward, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,594,879, 3.8.26. Appl., 22.11.19; cf. U.S.P. 1,477,113, B., 1924, 200).—By leading chlorine gas into an agitated mixture of allyl chloride and water below 20°, two isomeric glycerol dichlorohydrins are formed in satisfactory yield. It is of advantage to neutralise from time to time the hydrochloric acid formed. When the water present has become saturated with the dichlorohydrins these separate and may be recovered directly. On heating with sodium carbonate solution they afford glycerol in over 90% yield. T. S. Wheeler.

2 : 4-Dihydroxydiphenylethane. E. Klarmann, Assr. to Lehn & Fink, Inc. (U.S.P. 1,596,613, 17.8.26. Appl., 15.12.25).—Resorcinol and phenylacetonitrile are condensed in dry ether by the action of hydrogen chloride in presence of zinc chloride to 2 : 4-*dihydroxyphenyl benzyl ketimine hydrochloride*,

$$C_6H_3(OH)_2\cdot C(NH)\cdot CH_2Ph,HCl,$$

which on boiling with water gives 2 : 4-*dihydroxyphenyl benzyl ketone*, m.p. 121°, and this on reduction with zinc and hydrochloric acid yields 2 : 4-*dihydroxydiphenylethane*, m.p. 131°. It is a valuable non-toxic internal antiseptic especially as regards *B. typhosus*. T. S. Wheeler.

Manufacture of compounds from 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone and halogenated alcohols or their esters with carbamic acid. W. Carpmael. From I. G. Farbenind. A.-G. (E.P. 257,816, 1.2.26).—Halogenated aliphatic monohydric alcohols and their carbamates react with equimolar proportions of 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone, on fusion or on mixing solutions of the components, yielding compounds which are analgesics. Trichlorobutyl alcohol yields a *compound* of m.p. 61—63°, and trichloroethyl carbamate a *compound* of m.p. 75—76°. B. Fullman.

Manufacture of colourless products of [the reaction of] dialkylbarbituric acids with 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone. Chem. Fabr. auf Actien (vorm. E. Schering) (E.P. 255,434, 26.6.26. Conv., 16.7.25).—The yellow colour of the products obtained on melting together at 110—115° 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone with *CC*-dialkylbarbituric acids (such as diethyl- or *iso*propylallyl-barbituric acid) is due to atmospheric oxidation of the pyrazolone. White products are obtained by carrying out the melting and subsequent cooling in presence of an inert gas, such as carbon dioxide, hydrogen, nitrogen, etc. B. Fullman.

Pharmaceutical product [analgesic]. W. Schulemann and K. Meisenburg, Assrs. to Winthrop Chem. Co. (U.S.P. 1,596,769, 17.8.26. Appl., 15.5.25).—Δ^1-*cyclo*Hexenylethylbarbituric acid and 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone when mixed in ether or melted together, form a *compound*, m.p. 134—135°, which has strong analgesic and soporific properties. T. S. Wheeler.

Preparation of substituted allophanic alkyl esters. E. Merck, Assees. of C. Diehl (G.P. 427,417, 14.6.24).—Esters of the type : $NHR_1\cdot C(X) : N(R)\cdot CO_2\cdot alkyl$ (R = H or alkyl; R_1 = alkyl or aryl; X = O or S), are prepared by the action of the corresponding carbamides or thiocarbamides on neutral alkyl carbonates and sodium alkoxide, under slight pressure if necessary. For example, monomethylcarbamide is heated with diethyl carbonate and sodium ethoxide for 2 hrs. under pressure at 80°, the alcohol distilled off, the residue dissolved in water and acidified faintly with mineral acid, when *ethyl* N : ω-*methylallophanate*, m.p. 136—137°, separates in a pure state. The following esters are prepared similarly from the appropriate components : *methyl* N : ω-*methylallophanate*, m.p. 163°; *ethyl* N : ω-*dimethylallophanate*, m.p. 77—80°; *ethyl* N : ω-*allylallophanate*, m.p. 69—70°; *ethyl* N : ω-*phenylallophanate*, m.p. 105°; *ethyl* N : ω-*allylthioallophanate*, m.p. 47—49°. A. Davidson.

Production of tetrazoles. K. F. SCHMIDT (E.P. 257,418, 21.9.25. Addn. to 252,460; cf. U.S.P. 1,564,631, B., 1926, 216).—Carbonyl compounds, when treated with 2 mols. of hydrazoic acid in the presence of concentrated mineral acid, yield tetrazoles; if 3 mols. of hydrazoic acid are used, *C*-aminotetrazoles are formed. For example, *cyclo*hexanone with 2·5 mols. of hydrazoic acid yields 70% of tetrazoles, including (?) 1 : 5-*pentamethylene*-1 : 2 : 3 : 4-*tetrazole*, acetone with 2 mols. of hydrazoic acid yields 1 : 5-*dimethyl*-1 : 2 : 3 : 4-*tetrazole*, m.p. 71°; and benzophenone with 3 mols. of hydrazoic acid yields 5-phenylamino-1-phenyl-1 : 2 : 3 : 4-tetrazole, identical with phenyliminophenyltetrazolone (of A., 1900, i., 415).
B. FULLMAN.

Arsenic compounds [arsenoxides] of the aromatic series. A. J. RANSFORD. From L. CASSELLA & Co., G.M.B.H. (E.P. 257,361, 9.6.25).—1 : 2-Dihydrobenzoxazolone arsenoxides, having therapeutic value in spirochætal infections, are obtained by reducing the corresponding arsenic acids with hydriodic acid and sulphur dioxide. The following are described: 1 : 2-*dihydrobenzoxazolone*-5-*arsenoxide*; 4-*methyl*-1 : 2-*dihydrobenzoxazolone*-5-*arsenoxide*; 1 : 2-*dihydrobenzoxazolone*-4-*arsenoxide*; and 6-*chloro*-1 : 2-*dihydrobenzoxazolone*-4-*arsenoxide*.
B. FULLMAN.

Production of 3-chloro-5-amino-4-hydroxyphenyl-1-arsinic acid. I. G. FARBENIND. A.-G., Assees. of L. BENDA and W. SCHMIDT (U.S.P. 1,595,498, 10.8.26. Appl., 23.12.25. Conv., 2.3.25).—3-Chloro-4-hydroxyphenylarsinic acid is nitrated to the 5-nitro-compound, which is reduced and treated with acetic anhydride to yield the acetamido-derivative, and this, on hydrolysis with 16% aqueous caustic soda at 100°, gives 3-*chloro*-5-*amino*-4-*hydroxyphenylarsinic* acid, which is of therapeutic value and is unaffected by light.
T. S. WHEELER.

Process of making camphor. H. D. GIBBS and A. W. FRANCIS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,597,877, 31.8.26. Appl., 23.11.20).—A mixture containing 1 g. of *iso*borneol (as vapour) per 0·5—10 litres of air (measured at 20° and 760 mm.) is subjected to the action of a catalyst kept at 200—600°
B. FULLMAN.

Manufacture of esters of borneol and *iso*borneol. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 250,551 and 257,275, 12.3.26. Conv., 11.4.25).—(A) Camphene and a fatty acid are heated together at atmospheric pressure, first at about 100°, rising to about 150°, the formation of esters of borneol and *iso*borneol occurring quickly and completely. Especially when the acid is anhydrous esters of high m.p. are obtained in improved yield. For example, when 136 pts. of camphene are heated with 58 pts. of anhydrous formic acid, the reaction product contains 98% of ester. (B) If a considerable excess of fatty acid is used the reaction takes place at the ordinary temperatures.
B. FULLMAN.

Manufacture of synthetic camphor [conversion of bornyl chloride into camphene]. G. H. DUPONT and G. BRUS (E.P. 248,404, 2.3.26. Conv., 2.3.25).—Crude or pure bornyl chloride (pinene hydrochloride) is converted into camphene by treatment with metal resinates dissolved in excess of resin, at about 180°, a slightly volatile solvent, *e.g.*, resin oil, paraffin, etc., being added if necessary. The camphene is removed by steam-distillation, and separated from bornyl chloride by vacuum distillation. The residual resinic acid, if necessary after washing out the metal chloride formed, may be used again, or in the preparation of oil of resin, resin soap, etc. All the operations may be effected in the same vat provided with an agitator and reflux condenser. For example, 43 g. of dry sodium carbonate are added to 604 g. of colophony at 180—200°, 65 g. of bornyl chloride are added, and the mixture is heated for 2—3 hrs. at 180° under reflux, yielding finally 80% of camphene. Instead of pure bornyl chloride, the crude reaction product of pinene and hydrogen chloride may be used.
B. FULLMAN.

Extraction of the active biological principles from the ether extract of male fern. F. FUMAROLA (E.P. 256,768, 17.7.25).—A mixture of 1 pt. by weight of the ether extract with, *e.g.*, 4 pts. of magnesium oxide is digested with about 6 times its weight of distilled water at 40°, and filtered after about 5 hrs. The solution is acidified with dilute hydrochloric acid, and the precipitate is removed, dried below 40°, and treated for a few days with a mixture of, *e.g.*, 60 pts. of carbon tetrachloride and 40 pts. of ether, after which the residual silicic acid is filtered off, washed with a similar mixture until the wash liquor is colourless, dried at 40—80°, and desiccated on glass plates.
L. A. COLES.

Preparation of colloidal water-soluble bismuth for injection purposes. E. MERCK (G.P. 427,669, 27.10.22).—An alkaline bismuth solution is reduced, in presence of protective colloids, with sodium hyposulphite, air being excluded and a high temperature avoided. The colloid is precipitated by alcohol or a similar precipitant, and is carefully dried. For example, potassium sodium tartrate is dissolved in water and sodium hydroxide, and, while warming, basic bismuth nitrate is added gradually. To the solution, diluted with water, is added a 9% gum solution, sodium hydroxide, and an aqueous solution of sodium hyposulphite. After shaking for 1 hr. in a closed vessel a sol is obtained which is stable for a day in air. To obtain the gel, 87% alcohol is added to the solution, the precipitate washed four times with 58% alcohol, and dried by spreading on glass plates. The water-soluble gel contains about 40% of bismuth and 44% of organic matter. On injection, the product has no corrosive action on living tissue.
A. DAVIDSON.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Relation between intermittent and non-intermittent sector-wheel photographic exposures. R. DAVIS (U.S. Bur. of Standards, Sci. Papers No. 528,

1926, **21**, 95—139).—The difference between intermittent and equal non-intermittent exposures varies with the emulsion used, the illumination, the number of interruptions, and the rest periods between. When the illumination intensity is above a certain level, a greater effect results from the intermittent exposure. For lower intensities a loss is obtained. The gain or loss is accentuated by increased intermittence. The results were confirmed for six different types of emulsion. In explanation it is suggested that after extinguishing the illumination the latent image is subject to modification by two opposing influences, one tending to intensify it, the other to fade it; both decrease rapidly with time. At higher illumination values the resultant effect is a growth in density; at lower values, fading results. It is suggested that the reaction between light and the silver halide is not a single step, but that an intermediate condition exists between the first effects of light and the latent image proper. The first product of the reaction is in a nascent form, which, under the continued action of light, completes the reaction and forms the latent image. If the light is extinguished during this process, the nascent product, in the dark, partly returns to its former state, and partly completes the reaction, the degree of each depending on the illumination used. W. Clark.

Mordanting of silver images by cupric thiocyanate. A. and L. Lumière and A. Seyewetz (Bull. Soc. chim., 1926, [iv], **39**, 1246—1249).—In the Christensen mordanting bath containing copper sulphate, alkali thiocyanate, potassium citrate, and acetic acid, the active agent is cupric thiocyanate, which acts as well in simple aqueous solution as in presence of potassium citrate and acetic acid. The addition of these two substances increases the solubility of the cupric salt and the stability of the solutions. The white precipitate which slowly forms in the mordanting solution is cuprous thiocyanate. The mordanted image appears to consist of a complex of cuprous and silver thiocyanates. The composition is variable, but in general the substance contains 1 mol. of copper salt to 2 mols. of silver salt. Finely divided free silver, in the absence of gelatin, does not fix copper when shaken in the mordant bath. W. Clark.

Recovery of silver from exhausted fixing baths. J. I. Crabtree and J. F. Ross (Brit. J. Phot., 1926, **73**, 522—526).—The recovery of silver on a large scale from exhausted fixing baths by means of sulphide, zinc, sodium hyposulphite, and electrolytic methods is discussed. For large-scale use, precipitation with sodium sulphide is the most economical method. Precipitation with zinc dust is less rapid, but it is efficient and has the advantage that no hydrogen sulphide is formed. Although restoration of a fixing bath by de-silvering is possible, it is just as economical, and far preferable, to use a fresh bath. To revive a bath after de-silvering, it is necessary to add a further amount of hardener equal to that originally present, and also a quantity of thiosulphate equal to 50% of the original amount used. The resulting bath has approximately three-quarters the life of a fresh bath. W. Clark.

XXII.—EXPLOSIVES; MATCHES.

Metalammines and hydrazinates of the bivalent heavy metal chlorates and perchlorates as primary explosives.—W. F. Friederich and P. Vervoorst (Z. ges. Schiess- u. Sprengstoffw., 1926, **21**, 49—52, 65—69, 84—97, 103—105, 123—125, 143—146).—The metalammines of the chlorates and perchlorates of copper, cadmium, cobalt, zinc, and nickel are sensitive explosives, but only tetramminocupric chloride is suitable for use as a primary explosive. Their energy contents and detonation velocities are intermediate between those of mercury fulminate or lead azide and those of tetranitroaniline, T.N.T., or tetryl. The ammine chlorate compounds are considerably more sensitive than the corresponding perchlorate compounds, but cannot as a rule be used in technology as they lose ammonia rapidly and hydrolyse in damp air. The metalammines were prepared by saturating the cooled alcoholic solution of the metal chlorate or perchlorate with gaseous ammonia. The hydrazinates were obtained by cooling the aqueous or alcoholic solutions of the chlorate or perchlorate of the metal, and adding an aqueous or alcoholic solution of hydrazine hydrate with stirring, when the hydrazinate was precipitated. The *hydrazinates* of nickel chlorate and perchlorate and of cadmium perchlorate have not hitherto been prepared. The formulæ assigned to them are $Ni(ClO_3)_2,3N_2H_4$, $Ni(ClO_4)_2,NiClO_4\cdot OH,5N_2H_4,3H_2O$, and $Cd(ClO_4)_2,Cd(OH)_2,3N_2H_4,2H_2O$ respectively. The hydrazinates of the chlorates are very sensitive primary explosives, and surpass mercury fulminate and lead azide in brisance. The explosion points of some are below 100°. The perchlorate hydrazinates are less sensitive. The explosive properties of all metalammines and hydrazinates examined are summarised in a table which gives their formulæ, explosion points, sensitiveness to shock in the fall-hammer test, Trauzl lead block values, and ability to initiate explosion in tetryl, T.N.T., and trinitroanisole. S. Binning.

Apparatus for routine testing of the stability of a smokeless powder by determining the loss in weight [at 115°]. J. von Meerscheidt-Hüllessem (Z. ges. Schiess- u. Sprengstoffw., 1926, **21**, 137—141).—In testing the stability of a smokeless powder the hot-storage test and the method in which the loss of weight of the powder when heated at a definite temperature is determined give more reliable results than any other. The loss-of-weight method has the disadvantage that it requires considerable time, and is therefore impracticable when many samples require to be tested. An apparatus, drawings of which are given, has therefore been designed that enables 37 samples to be tested in 8 days of 12 hrs., or if a duplicate apparatus is available 74 samples in 16 days of 6 hrs. It consists of a bath which supports a frame capable of taking 38 test-tubes, which are immersed in the heating medium consisting of an aqueous glycerin solution boiling at 115°, the temperature chosen for the test. The apparatus also includes means for raising the frame with its test-tubes out of the bath, and lowering it into a box where the samples cool to room temperature in an atmosphere free from moisture, after which the loss of weight is determined. S. Binning.

PATENT.

Stabiliser for nitrated organic compounds [explosives]. W. A. PATRICK, Assr. to SILICA GEL CORP. (U.S.P. 1,596,622, 17.8.26. Appl., 18.2.21).—Silica gel of the type described in U.S.P. 1,297,724 (B., 1919, 363 A), when mixed with organic nitro-compounds which are used as explosives, *e.g.*, nitroglycerin or nitrocellulose, rapidly absorbs any oxides of nitrogen formed and stabilises the explosive more efficiently than the chemical substances hitherto employed. Its use in dynamite renders unnecessary the inclusion of anhydrous copper sulphate hitherto employed as an absorbent for moisture. T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Chemical and bacteriological examination of the London waters for the twelve months ended March 31, 1926. A. C. HOUSTON (Metropolitan Water Board, 29th Ann. Rept., 1926, 118 pp.; cf. B., 1925, 145, 939).—Chlorination of Thames river water has been continued throughout the year with marked success, some 24,540 million gallons of raw water being treated with 56·21 and 23·26 tons of bleaching powder and liquid chlorine respectively; treatment of the raw water in this manner, and allowing it to flow by gravity to the filters instead of first pumping it into the Staines reservoirs, has resulted in a net saving of £10,056. The average dose of chlorine applied was 0·4 p.p.m., and the average cost works out at 9·8d. per million gals. Of the samples taken of the chlorinated water 27·4% contained *B. coli* in 10 c.c., as compared with 41·2% of the samples taken of the Staines stored water during the period 1908—16. Chlorine was applied to New River water on 137 days during the winter months, 4490·7 million gals. of water being treated with an average dose of 0·283 p.p.m. of chlorine at an average cost of 6·5d. per million gals. treated. Permanganate, average dose 0·205 p.p.m., was added on 103 days at a cost of 11·8d. per million gals., and on 34 days ammonium chloride, instead of permanganate, was used as taste preventer. In December taste troubles, lasting for one or two days only, were encountered for the first time since chlorination was commenced in 1919. When the taste was noticed the dose of chlorine was reduced and, as an extra precaution, for a period of several days the dose of permanganate was doubled and ammonia added as well. For a period of 4 months, with intermissions, permanganate and ammonia treatments were alternated, with the object of comparing the two as regards sterilisation effect. The dose of permanganate applied was 0·2 p.p.m. and that of ammonia 0·2 p.p.m. in terms of nitrogen. The results were slightly in favour of chlorine plus ammonia. Other experiments, in which ammonia was added to water prior to chlorination, showed that the addition of ammonia increases the bactericidal effect of the chlorine, but more time is required to obtain the full sterilising effect. "Dichloramine" methods of treatment, consisting of the addition of a "mixture" of chlorine and ammonia to water in bulk, gave encouraging results. *Leptospiras* were found to be present in all raw sources of supply, and also in many filtered waters and pure deep-well waters. All the cultures made were proved to be non-pathogenic, except in one instance (Deptford [Garden] Well), and this supply has since been chlorinated as a precautionary measure. The doses of chlorine used for the destruction of *B. coli* more than suffice for the death of *Leptospiras*. The experiments at Barn Elms, in which water is treated by rapid filtration methods prior to treatment on slow sand filters, have demonstrated that water so pretreated can be satisfactorily dealt with by slow sand filters at from 3 to 4 times the normal rate. W. T. LOCKETT.

Determination of organic matter in water by means of potassium dichromate and sulphuric acid. W. E. ADENEY and (MISS) B. B. DAWSON (Sci. Proc. Roy. Dublin Soc., 1926, **18**, 199—202).—The water (50 c.c.) is concentrated to 5 c.c. with a little sulphuric acid to remove chlorides, mixed with sulphuric acid (50 c.c., 50% by vol.) and 0·25*N*-potassium dichromate (50 c.c.), heated at 100—110° for 2 hrs., and the excess of oxidant then titrated with standard ferrous sulphate. The organic matter is oxidised almost completely to carbon dioxide, water, and ammonia. G. M. BENNETT.

Rate and mechanism of aeration of water under open-air conditions. W. E. ADENEY (Sci. Proc. Roy. Dublin Soc., 1926, **18**, 211—217; cf. A., 1920, ii, 686; B., 1923, 781 A).—Further experiments confirm the views already published. G. M. BENNETT.

Determination of carbon monoxide in the air of workshops. F. SCHOOFS (J. State Med., 1926, **34**, 575—577).—During the removal of oxygen by pyrogallol, a considerable excess of alkali must be present to prevent the formation of carbon monoxide. (Cf. Drakeley and Nicol, B., 1925, 944.) F. R. ENNOS.

Bactericidal action of cadmium compounds. E. A. COOPER and (MISS) L. I. ROBINSON (J.S.C.I., 1926, **45**, 321—323 T).—Cadmium compounds are active germicides, being more efficacious than the salts of many metals, with the exception of silver and mercury. Cadmium salts are much more powerful disinfectants than the corresponding zinc compounds, and approach mercuric salts in germicidal power, although chemically cadmium is more closely allied to zinc. Organic derivatives of cadmium tend to be less effective as germicides than the inorganic salts. Solutions of cadmium hydroxide in ethylenediamine, however, exert a strong bactericidal action.

Insecticidal value of certain chemicals. BRINLEY. —See XVI.

PATENTS.

Preparation of chlorine water [for sterilising sewage]. I. G. FARBENIND. A.-G., Assees. of P. SIEDLER and W. MOSCHEL (G.P. 429,037, 13.7.22).—The chlorine water is made in an apparatus arranged to permit the flow of water to be kept continuously in contact with pure chlorine under slight pressure. W. G. CAREY.

Insecticides. E. C. R. MARKS. From RUBBER SERVICE LABORATORIES Co. (E.P. 257,644, 29.4.25).—See U.S.P. 1,573,490; B., 1926, 379.

Insecticidal and fungicidal media (E.P. 257,171).— See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOVEMBER 26, 1926.

I.—GENERAL; PLANT; MACHINERY.

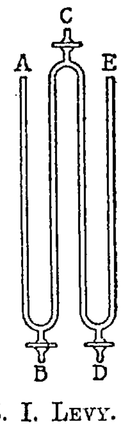
Apparatus for the automatic regulation of furnaces. VERNEY (Compt. rend., 1926, **183**, 561—562).—By means of two insulated copper tubes, mounted on the same axis in the form of a horizontal arch, contact may be made with a strip of malleable copper previously bent in the shape of the time–temperature curve required. The vertical stand which supports the copper strip is moved regularly in the direction of its plane, and the strip thus makes contact with one of the copper tubes. An endless screw which is attached to the torsion head of a galvanometer and is actuated by means of an electric motor, brings back the insulated portion of the arched contact on to the copper strip again. The galvanometer thus indicates the temperature corresponding to the ordinate of the curve, and sets in action a relay which adjusts the temperature of the furnace. J. GRANT.

Recent work on the measurement and industrial importance of particle size. J. PARRISH (J. Oil and Colour Chem. Assoc., 1926, **9**, 252—259).—A general survey is given of the literature on particle size, including methods of its measurement and its importance in relation to the paint, rubber, paper, and cement industries. D. F. TWISS.

Value of a direct measurement method for particle size determination. H. GREEN (J. Oil and Colour Chem. Assoc., 1926, **9**, 226—234).—A direct or photomicrographic method for the measurement of particle size has the value that it gives a "particle-distribution" curve showing the relative frequency of occurrence of various diameters. Such a method also does not always involve the necessity to assume that the particles are cubical or spherical. The particle distribution curve also enables a calculation as to the particular average diameter needed for special work. With certain clays and the so-called inert powders especial care is required to ensure that the sample examined is truly representative. D. F. TWISS.

Apparatus for the separation of grit and coarse particles from fine powders. G. GALLIE and B. D. PORRITT (J. Oil and Colour Chem. Assoc., 1926, **9**, 235—238).—The apparatus consists of a metal funnel terminating in a removable cup, 25 mm. in diameter, the bottom of which is formed of wire gauze of any desired mesh. A weighed quantity of the powder is introduced into the funnel, and water (filtered through metal gauze) is added until the funnel is approximately half filled. A jet of filtered water issuing from a tube reaching almost to the bottom of the funnel, and arranged so that its distance from the gauze bottom is adjustable, forces the particles through the sieve and disintegrates loose agglomerates. The residue in the cup is finally washed, the cup being then removed and dried at 100° preparatory to weighing. D. F. TWISS.

Apparatus for determination of specific gravity of liquids. A. N. N. TRENITÉ (Chem. Weekblad, 1926, **23**, 465).—The apparatus consists of a glass tube, A B C D E, 12 mm. in diameter, bent through 180° at B, C, and D so as to form two U-tubes, A B C, E D C, connected at C; stop-cocks are fused on at B, C, and D. Each of the four vertical limbs so formed is provided with a millimetre scale. Water is introduced through the limb, A, and the fluid to be tested through the limb, E, thus enclosing air in the inner limbs and the bend, C. The differences in height of the two water levels in A B C and of the two fluid levels in E D C give the specific gravity directly. S. I. LEVY.



PATENTS.

Open-fire kiln. P. A. MEEHAN, Assr. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,596,502, 17.8.26. Appl., 21.8.24).—Door-locking devices for the exit end of a continuous tunnel kiln are described. R. B. CLARKE.

Cylindrical dryer for low-temperature drying. J. BRABAEK (G.P. 431,564, 3.10.22).—Materials for which high temperatures are unsuitable are dried by a dry air blast in a cylinder around the upper surface of which are a large number of nozzles arranged radially, and connected with an air pressure main. W. G. CAREY.

Heating, humidifying, and evaporating apparatus. R. J. KRAUSE (E.P. 257,468, 10.12.25).—The apparatus comprises a number of rotating concentric drums supported on a disc-like driver; alternate drums project further from the driver than the others, and the projecting drums have spaces left between them and the driver, so that when material is supplied to the drum nearest the axis it will spread over the interior surfaces of all the drums in succession, being held inside each drum by centrifugal force. Heating means such as electric resistances may be applied to the exterior surfaces of the drums. B. M. VENABLES.

Centrifugal evaporation. M. J. KERMER (U.S.P. 1,598,282, 31.8.26. Appl., 16.1.25).—The stationary housing round a rotating cylinder forms a steam chamber, and is provided with an annular chamber which contains fluid as a seal. S. BINNING.

Centrifugal separators. C. S. GARLAND, J. W. HINCHLEY, and C. S. WATSON (E.P. 255,914, 4.2.25).—

A centrifugal separator designed for the continuous discharge of both separated constituents, which may be a solid and a liquid or heavier and lighter liquids, is constructed with two conical bowls, 5, 6, with their bases together (see Fig. 1). The lower bowl is rotated by a rigid connexion to the shaft and the upper bowl by a resilient connexion combined with a screw thread on the shaft so that when the upper bowl lags behind the lower the common bases of the bowls separate slightly and allow the collected solid matter (or heavier liquid) to fly out. The lag of the upper bowl is produced by friction between a fixed surface, such as a flanged sleeve, 11, and the accumulated solid matter. The lighter constituent discharges over an internal rim in the usual way. Alternatively the upper bowl, 5, may not rotate except slightly on a fixed screw thread (see Fig. 2); the whole of the upper bowl then forms the frictional surface, and is held in the closed position by fixed external springs, 43, easily accessible for adjustment.

B. M. VENABLES.

FIG. 1.

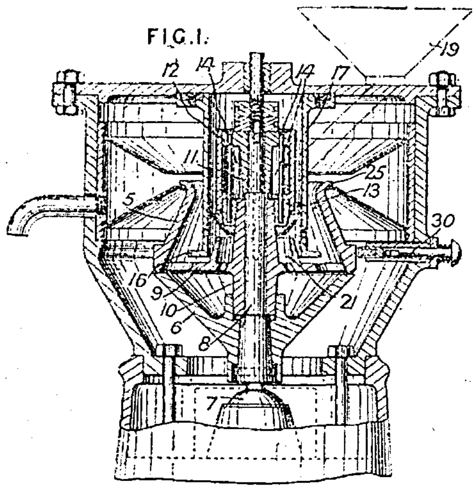
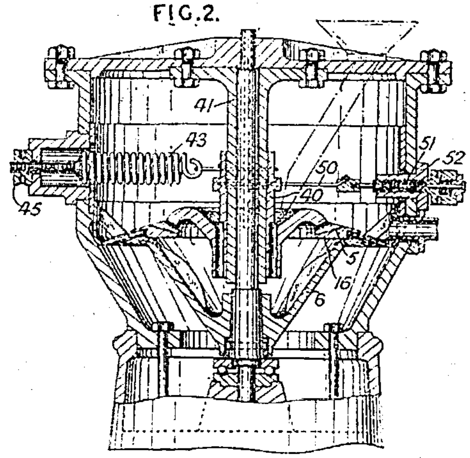


FIG. 2.



Centrifugal separating machines. R. A. STURGEON (E.P. 257,422, 1.10.25).—A centrifugal separator is provided with means for removing separated solid matter without stopping the rotation comprising a pair of pistons and a cap vertically fixed but rotating with the hollow vertical shaft of the machine. Embracing the pistons is a cylinder, also rotating, which can move vertically, and is divided into three compartments. The uppermost compartment forms the collecting surface for solids, and is closed when the cylinder is raised against the cap and opened for discharge of solids when lowered. The middle compartment is filled with water to raise the cylinder, the lowest is filled with water to lower it. The water for operation is supplied to the appropriate compartment under no particular pressure, the pressure for operation being obtained by the rotation of the apparatus.

B. M. VENABLES.

Treatment of solid materials with liquid reagents. [Preparation of aluminium sulphate from bauxite etc.] H. N. SPICER, and DORR Co. (E.P. 257,643, 28.4.25). —A continuous process of leaching in a series of agitators which are so arranged that the coarser particles are retained sufficiently long so that they do not overflow until they have been reduced to fine particles by the action of the solvent. The strong solution is recovered, and the fine particles are washed by decantation in a series of thickeners. The process is particularly suitable for the preparation of aluminium sulphate solution of high concentration from bauxite and sulphuric acid. The bauxite, acid, and part of the overflow from the second thickener are mixed in the first agitator, the strong aluminium sulphate solution overflows from the first thickener, and water is added to the last thickener. Instructions are given for calculating the size of the thickeners, viz.: For clear overflow, free falling rate of particles multiplied by area of thickener = volume rate of overflow. For thickest possible underflow, the necessary hindered settling period multiplied by volume rate of underflow = volume of thickener, which divided by the area already calculated gives the necessary height. Since heating increases the efficiency of the thickeners the incoming liquids or pulp may be heated externally to the thickeners, but not in them on account of convection currents.

B. M. VENABLES.

Liquefaction and separation of gaseous mixtures. R. C. E. MEWES (E.P. 257,325, 25.5.25).—A mixture of liquefied gases such as air is introduced simultaneously into two rectifying stills, one of which operates under moderate pressure and the other at nearly atmospheric pressure. The low-boiling constituent obtained from a preliminary rectification in the pressure column is used both to heat the still of the atmospheric column and, after reduction of pressure, as washing liquid in the same column, together with a portion of the low-boiling liquid obtained from the bottom of the atmospheric column and that used to heat the still of the pressure column. The cold to make up losses is provided by low-boiling constituent (or air) supplied under high pressure to the still of the pressure column.

B. M. VENABLES.

Refrigerating machines for absorption of ammonia or any other substance having the same properties. A. E. ANDERSON, G. MAIURI, and R. F. BOSSINI (E.P. 257,458, 21.11.25).—In an absorption refrigerating apparatus the inert carrier gas is caused to circulate by thermo-siphon effect, the flow side being heated in the boiler by vapour therefrom, or in a separate

heater. The return is cooled in the condenser. Heat interchange may be provided between the gas arriving at and leaving the evaporator, also between the liquor to and from the boiler. B. M. Venables.

Purification of liquids by adsorption. P. W. Prutzman and A. D. Bennison, Assrs. to General Petroleum Corp. (U.S.P. 1,598,254—6, 31.8.26. Appl., [a] 17.8.25, [b] 23.3.26, [c] 23.3.26).—Liquids are purified by treatment with the following finely divided substances: (a) natural magnesium silicate, (b) magnesium silicate after treatment with hydrochloric acid, or (c) magnesium silicate after treatment with sulphuric acid. S. Binning.

Roll or cylinder mills for grinding paint and other substances. A., O., and W. Buhler (Buhler Bros) (E.P. 257,727, 18.8.25).—A number of rolls are arranged over each other, but the axes are not in the same vertical plane, and the circumferential distance between the lines of contact of adjacent rolls is less than one-third of the circumference of a roll. The velocity ratio between adjacent rolls lies between 3·2 : 1 and 2 : 1. With this arrangement the peripheral speed of the fastest roll may be higher than 750 ft./min., the ordinary limit for paint. Arrangements are described for very fine adjustment of the rolls, and feed guides are provided so that the material is spread over the whole length of the rolls, any excess working over the ends being caught in other guides. B. M. Venables.

Pulverising apparatus. R. J. Tugwood. From Jeffrey Manuf. Co. (E.P. 257,665, 4.6.25).—A disintegrator is provided with yielding hammer-pieces in which two holes are formed, and a pin passes through each hole connecting the hammer with the driving discs or spider. One hole is circular, the other is a curved slot, so that when the hammer hits an unbreakable obstruction it may turn aside. The hammers may be held in their operative position entirely by centrifugal force, or, in addition, a thin, brittle, and easily renewable piece of metal may be placed across the slotted hole to prevent the movement of the hammer. B. M. Venables.

Furnaces. O. Y. Imray. From T. E. Murray (E.P. 258,760, 23.12.25).

Furnaces. J. Woods (E.P. 258,347, 17.6.25).

Bearings for centrifugal extractors. W. H. Sumbling (E.P. 246,153, 14.1.26. Conv., 15.1.25).

Safety devices for centrifugal machines. T. Broadbent & Sons, Ltd., and H. Broadbent (E.P. 258,789, 6.3.26).

Air filters. National Air Filter Co., Assees. of H. E. Birkholz (E.P. 249,851, 16.3.26. Conv., 30.3.25).

Devices for separating liquids of different specific gravities. E. W. Green (E.P. 258,662, 24.6.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Application of *X*-rays to the laboratory jig-testing of coal. C. N. Kemp (Proc. S. Wales Inst. Eng., 1926, **42**, 411—437).—A method is explained for testing in the laboratory samples of coal from certain aspects of its ash content. The apparatus consists essentially of a U-tube of square cross-section; near the bottom of one limb is a grid upon which, and nearly filling the limb, the sample of coal under test rests. The U-tube is nearly filled with water, to which rhythmic impulses are imparted by the action of compressed air at the top of the second limb, causing it to surge to and fro through the coal sample. The coal sample is observed by the passage through it of *X*-rays, by means of which the separation of the adventitious ash may be followed. Exhaustive separation of this matter may be effected. The relative depths of the coal (which shows light) and of the ash (which shows dark) give a key to the proportions of them in the raw coal. The test may be used to show the amount of dirt in the coal, and whether a sample of washed coal can be improved by further washing, or whether washing has been carried too far in that the dirt contains "clean coal." Hence it may serve to control washing processes and to determine the relative values of samples of coal. R. A. A. Taylor.

Volatile matter in coke. G. E. Foxwell (Gas World, 1926, **85**, Coking Sect., 102—105).—From a survey of the experimental researches of Porter and Ovitz and of Burgess and Wheeler, the advisability of expelling the whole of the volatile matter from coke is discussed. Every 1% of volatile matter left in the coke corresponds to a deficiency of 4 therms in the gas per ton of coal carbonised. The expulsion of 1% of volatile matter requires 1 therm of heating gas and a further hour's heating in the oven. The by-products remaining in high-temperature coke are of no value. It is concluded that for coke-oven plants having no sale for their gas the last traces of gas are not worth the additional time, wear and tear necessary; for gas works, the therms in the gas are of more importance, consonant with the production of good coke. A. C. Monkhouse.

Coking capacity of coal. D. J. W. Kreulen (Chem. Weekblad, 1926, **23**, 449—453; cf. B., 1925, 160).—Mixtures of the same coal reduced to different particle sizes with varying proportions of sand and of graphite were coked under exactly the same conditions, and the resistances to impact of the resulting coke examined. The resistance is greater the smaller the size of the particles of added indifferent material, and reaches a maximum in all cases with addition of about equal total surfaces of added material. The cokes containing sand show much lower resistances than those containing graphite. The proportion of non-coking coal which may be added to a coking coal to form a good coke may be increased by finer division of the former. S. I. Levy.

Low temperature carbonisation: the economic side. O. J. Parker (Proc. S. Wales Inst. Eng., 1926, **42**, 403—410).—Attention is directed to the low-temperature carbonisation of lower-grade non-coking coals. The aim is to provide a smokeless coke for domestic use, together with other remunerative products. Mere increase in the calorific value of the coke over that of the raw fuel does not in itself constitute grounds for treating the raw fuel, especially as in the higher grades the increase goes down in proportion. Combustibility and robustness of the product must be considered. Graduated heating up of the charge is

advocated to enhance the yield and value of the tars, and to attain in them a partial fractional distillation. Briquetting is advocated for slacks, smalls, and washery slurries. Costs are quoted for a German (the Delkeskamp) plant meeting these requirements.

R. A. A. TAYLOR.

Value of coal carbonisation products and its determination in the laboratory. R. GEIPERT (Gas- u. Wasserfach, 1926, **69**, 861—862).—The coal sample (10 g.) is dropped, in the form of a briquette, through the vertical limb of a T-piece attached to the top of a silica tube placed vertically in a furnace maintained electrically at 1100°. The volatile carbonisation products pass out through the horizontal limb of the T-piece, through a short tube packed with glass wool to catch the tar, and the gas is collected over water in a graduated vessel. Three determinations should be made and can be completed within 1 hr., and the cal. value and composition of the united gas may be determined in the usual manner. From the value of the therms obtained, and of the coke and tar, the total value of these products per ton of coal is ascertained, and forms a basis for comparing different classes of coals. In determining the value of the coke, corrections are introduced for its ash content and mechanical quality, and account is taken, in the final valuation, of the moisture content of the original coal.

W. T. K. BRAUNHOLTZ.

Steaming in continuous vertical retorts. M. BARASH (Industrial Chemist, 1926, **2**, 351—355; cf. Fuel Res. Bd., Tech. Paper 8, B., 1923, 1163 A).—The history of the continuous vertical retort is reviewed with special reference to steaming. A series of tests is recorded in which the yield of gas per ton of coal carbonised was increased by additional steaming from 16,970 cu. ft. of calorific value 503 B.Th.U. per cub. ft., to 70,000 cub. ft. of calorific value of 350 B.Th.U. per cub. ft. All tests, including those in which the quality of the gas lay intermediate between these two values, gave results in agreement with theoretical calculations.

S. PEXTON.

Relationship between the calorific value of a gas and either the oxygen necessary to burn it, or its products of combustion. H. FAHRENHEIM (Gas- u. Wasserfach, 1926, **69**, 838—840).—Taking the water formed as steam, the volume of the products of combustion of unit volume of coal gas or coke-oven gas or mixtures of these bears a strikingly constant ratio to the gross calorific values of the respective gases. Owing to the condensation of the water after explosion, however, the volume of the products cannot be measured, but the oxygen used in the explosion bears similarly a ratio which is nearly constant to either the gross or nett calorific value. The amount of oxygen necessary to burn 100 c.c. of gas gives the gross calorific value when multiplied by 50·9, and the nett when multiplied by 45·85, these figures being the mean for a series of gases, and giving a result in kg.-cal. per cub. m. The greatest deviation from the calorific value calculated from the analysis is 1%. Extended to blast-furnace and producer gases not containing unsaturated hydrocarbons, similar relationships hold, the ratio of oxygen to nett calorific value being about 56·5; the deviations from one gas to another are greater, but the maximum error is only 1·2%. To find the gross calorific value, the nett has to be multiplied by a factor particular for each type of gas: blast-furnace gas, 1·03; producer gas, 1·06; blue water gas, 1·09.

R. A. A. TAYLOR.

Gas purification. H. BLAKELEY (Gas J., 1926, **175**, 478—480).—Purification of gas from hydrogen sulphide is reviewed from the plant manufacturer's point of view. In laying down new plant it is desirable for future developments, to provide two or three proportionately larger iron oxide purifiers than four smaller purifiers. When using purifiers with water lutes it is essential to prevent foul oxide from falling into the lutes and producing acidulated water which results in extensive corrosion of the side plates and covers. Also the valve system should be such that each purifier can be independently controlled and that any sequence of purifiers can be worked. The resistance of a purifying installation can be halved by dividing the oxide in each purifier into two shallow layers, and leading the gas from the intervening space through the two layers in parallel.

S. PEXTON.

Burkheiser process of gas purification. W. BURKHEISER (Gas- u. Wasserfach, 1926, **69**, 765—771).—The various stages in the evolution of the process are described. In the latest form the gas, after removal of tar, passes into a rotary scrubber containing a suspension of iron hydroxide and free sulphur where the gas is purified from cyanogen and hydrogen sulphide. The mixture in the purifier is pumped to an enclosed stirrer where it is mixed with ammonium sulphide (concentrated gas liquor) and the sulphur is dissolved. The mixture is then transferred to a closed settling tank where the ammonium polysulphide and thiocyanate are separated. The ammonium polysulphide is decomposed to ammonium sulphide and free sulphur, which latter is burnt to sulphur dioxide, as in the old process, and used for the recovery of the ammonia in the gas as ammonium sulphite. The iron sulphide from the settling tank is pumped to a rotary scrubber where, after admixture with freshly revivified material to remove any polysulphide, it is revivified with air and subsequently pumped back to the purifier. This last stage of development, viz., "wet" purification and "wet" revivification, awaits large-scale experiment.

A. C. MONKHOUSE.

Refrigeration of gas for the removal of naphthalene, water and ammonia. F. LENZE and RETTENMAIER (Gas- u. Wasserfach, 1926, **69**, 689—691).—Foul gas leaving a works condenser at 10—30° was further cooled in a refrigerating apparatus to 0°, where the naphthalene content was reduced from 13—26 to 1·7—2·6 grains per 100 cub. ft., and the ammonia content reduced from 75—175 to 30—45 grains per 100 cub. ft. with no measurable loss in the benzol content or the calorific value of the gas. The condensate from the refrigerator contained 9·6% of ammonia. The partial pressure of ammonia in equilibrium with ammonium carbonate solutions of varying strengths and also the freezing points of ammonium carbonate solutions were determined.

S. PEXTON.

Direct recovery of pure sulphur from gases containing hydrogen sulphide by means of solutions

of copper salts. F. Fischer and P. Dilthey (Brennstoff-Chem., 1926, 7, 300).—If a gas containing hydrogen sulphide is passed through a solution of cupric chloride in brine at a slightly elevated temperature a yellow crust of almost pure sulphur is obtained. The purity of the product increases with increasing concentration of the cupric chloride and with increasing temperature, suitable conditions being a 10% solution at 70—80°. The cuprous chloride simultaneously formed is converted back into cupric chloride by blowing air through the solution, oxidation being favoured by increased temperature and retarded by increased concentration.

W. T. K. Braunholtz.

Reduction and hydrogenation of carbon monoxide. F. Fischer and H. Tropsch (Brennstoff-Chem., 1926, 7, 299—300).—The formation of a small quantity of "synthol"-like products under certain conditions from water gas at atmospheric pressure, recorded by Elvins and Nash (cf. A., 1926, 917), had been previously observed by the present authors, though the production of hydrocarbons (cf. B., 1926, 475) can be so conducted as to eliminate this reaction, which, whilst proceeding to a considerable extent under pressure, is here only a subsidiary one. The theory that unstable carbides are intermediate in the formation of higher hydrocarbons at atmospheric pressure is reaffirmed.

W. T. K. Braunholtz.

Chemical changes in the "Berginisation" of coal. A. Spilker and K. Zerbe (Z. angew. Chem., 1926, 39, 1138—1143).—Treatment of naphthalene in an autoclave with hydrogen at 100 atm. (initial) and 470° results in transformation of about one-third into hydronaphthalenes, mostly tetralin. At lower temperatures and pressures less is attacked; at higher temperatures, hydrobenzenes and other decomposition products are formed. Similar results are obtained in presence of aluminium chloride, the yield of higher-boiling products being somewhat greater. Hydrogenation of anthracene at 450° and an initial pressure of 75—80 atm. yields a completely liquid product of mixed hydroanthracenes. Phenanthrene and acenaphthene under the same conditions yield 30 and 90%, respectively, of hydrogenated liquid mixtures. Fluorene yields 30% of liquid hydrofluorenes when treated at 465°; carbazole remains unattacked under the same conditions. S. I. Levy.

Conversion of lignin into humic acid and humin in the formation of humus and lignite from conifer remains. W. Grosskopf (Brennstoff-Chem., 1926, 7, 293—299).—A comparison of the chemical composition of successive stages in the conversion of green pine needles, through beds of dead needles and dry peat, into lignite. Pentosans were determined by conversion into furfuryl alcohol, and cellulose by chlorination. By extraction with cold, 42% hydrochloric acid lignin and humus remain undissolved, and of these the humus alone remains undissolved on treatment with acetyl bromide. In the series from fresh pine needles to lignite the content of pentosans shows a gradual but steady decrease, and the same appears to apply to hexosans; no appreciable formation of oxycellulose occurs during the production of humus. The content of cellulose also gradually diminishes, the substance being changed into a slack material having the properties of neither oxycellulose, humus, nor coal. Cellulose is absent from lignite. The rapid increase in the content of humus corresponds roughly with the rapid decrease in that of lignin, and indicates the conversion of the latter into the former, the change taking place in two stages, viz., the formation of humic acid reaching its maximum in dry peat, and the subsequent formation of humin. It is considered that humus beds composed of conifer remains constituted, in the Tertiary period, the beginnings of present-day lignites. W. T. K. Braunholtz.

Carbonisation of oil shale in rotary retort. E. Fleischmann (Brennstoff-Chem., 1926, 7, 229—232).—A Thyssen rotary retort of 80 tons per day capacity was heated by two rows each of 10 double-heating shafts, fired with a mixture of shale coke and shale. The ash is used in the cement manufacture. The shale to be carbonised passes through the retort in 2 hrs. The gases are scrubbed with heavy oil, which extracts the heavier fractions, and then cooled to 100°, purified with milk of lime, and debenzinised by a Linde refrigerator. The heat value of the shale was 100 cal. and of the shale coke 500 cal. per kg. One ton of shale yielded in practice 36·90 cub. m. of gas and 30·11 kg. of oil. The heat balance of the process and analyses of the products are given. H. Moore.

Cracking petroleum by the Dubbs process. F. Meyer (Brennstoff-Chem., 1926, 7, 232—235; cf. Dunstan and Pitkethly, B., 1925, 60).—A description of the Dubbs cracking process and comparison of the advantages of different methods of cracking. H. Moore.

Diesel oil. H. Kühl (Petroleum, 1926, 22, 977—983). —The water content of Diesel oils should not exceed 1%, the oil should still be liquid at 5°, or, in case of coal tar oil, nothing should separate out at 15°; in general, viscosity should not exceed 3° Engler at 20°. Flash point (Abel–Pensky) should lie between 55° and 150°. The coke residue should not exceed 8, 1, and 3·5% for crude mineral oil, gas oil or kerosene, and coal tar oil, respectively. The ash of crude or residue oils should not exceed 0·03—0·05%; amount insoluble in benzene or xylene should not exceed 0·3%. A hydrogen content of 11½%, or for tar oil 6½%, is minimum. A small content of sulphur is unimportant, but much is said to attack the exhaust system. Calorific value is a measure of the energy obtainable from the oil. For crude oil, 9800; gas oil, 10,000; residue oils, 9900; kerosene, 9600; coal-tar oil, 8600 kg.-cal. per kg. are minima. Of the crude oil, 20; gas oil, 60; residues, 20; tar oil, 75% at least should come over at 350°, whilst kerosene should give at least 50% at 300°. The oil should not contain more than 0·3% of mineral acid (as SO_3) nor more than 0·05% of chlorine. An oil which fails in some of these respects may still, however, be useful—experience alone can settle the point. The present tendency is to filter or centrifuge the ash and coking constituents out of mineral residue oils, with what success is still doubtful. The merits of oil from petroleum, lignite, and coal, and low-temperature carbonisation, and the probable duration of the petroleum supply, are discussed. W. N. Hoyte.

Activated charcoal for gasoline recovery. G. MÜLLER (Petroleum, 1926, 22, 1043—1047).—The quantity of steam used on the works scale in regenerating the charcoal is found to be less than that predicted from laboratory experiment and calculation, on account of the "rinsing" effect of the current of steam. The use of the compression method, in which the steam is passed through a heat exchanger, and serves to generate fresh steam under a partial vacuum, economises steam and prevents loss of gasoline. W. N. HOYTE.

Simple viscosimeter with scale [for oils]. R. VON DALLWITZ-WEGNER (Petroleum, 1926, 22, 1048—1049).—A worm rotating at constant speed in a cylindrical chamber, heated to the desired temperature and containing the oil under test, causes the oil to rise in a vertical glass tube, with scale, to a height depending on the viscosity of the oil. The scale can be divided in any of the usual units. W. N. HOYTE.

PATENTS.

[Coal] pulverising apparatus. W. R. WOOD. From INTERNATIONAL COMBUSTION ENGINEERING CORP. (E.P. 257,436, 16.10.25).—Pulverisers for coal are found to work better when the coal is slightly moist, but this moisture might seriously interfere with the subsequent handling of the finely powdered fuel; therefore the circulating carrier air on its passage from the pulveriser to the cyclone collector is relied on to dry the coal. On the return journey from the cyclone the air is cooled and the moisture condensed out of it, and it is then re-heated before re-entering the pulveriser. B. M. VENABLES.

Drying and low-temperature carbonisation of fuels. R. DRAWE (G.P. 432,251, 23.4.25. Addn. to G.P. 425,316).—The flues of the original patent (B., 1926, 573) have internal projections, affording a more efficient heat transfer from the heating gas to the material being heated. The inlets and outlets of the flues are so proportioned that part of the heating gas is passed through the material under treatment. W. T. K. BRAUNHOLTZ.

Coke-ovens. J. STEPHENSON (E.P. 244,772, 14.12.25. Conv., 20.12.24).—A regenerative oven is described (see figs.). When the flue at the left of the centre of the base is in connexion with the chimney, air enters the regenerators 26, 27, 29 and 28, and passes via 36 and 35 to the sole of the oven. Fuel gas is admitted to the combustion flues 23, at several points 41, and meets the hot air from the oven sole. The combustion completed, the products containing excess of secondary air are diverted at the centre of the setting to the top of the conjugate set of flues heating the other half of the oven face, where they pass downwards and meet further supplies of fuel gas. The waste gases pass through the regenerators to the main flue. Periodically the direction of flow of the gases is reversed. The arrangement of flues ensures a very uniform heating. A further feature is the flue 9 (Fig. 1), which connects with the oven at 11; this flue allows coal gas, otherwise wasted, to be collected during the charging of the ovens. S. PEXTON.

FIG. 1.

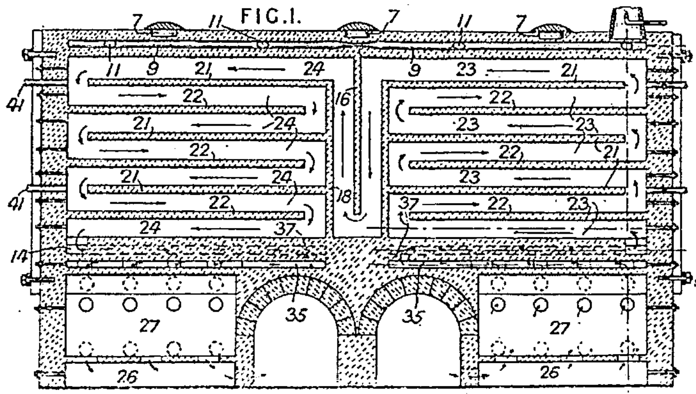
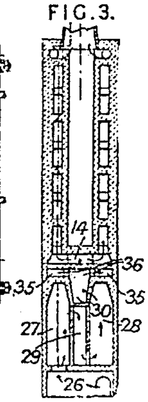


FIG. 3.



Gas-making and carbonising apparatus and process. W. W. ODELL (U.S.P. 1,598,217, 31.8.26. Appl., 17.12.23).—Fuel slowly gravitates through a vertical retort. Part of the fuel is burnt by the admission of air through a number of ports arranged around the retort at different levels. The fuel core, as it passes through the surrounding zone of combustion, is carbonised and the gaseous products are withdrawn through offtakes located within the fuel. S. PEXTON.

Utilising waste heat in the production of water-gas. BAMAG-MEGUIN A.-G. (E.P. 253,088, 11.5.26. Conv., 4.6.25).—It has become the general practice to utilise the water-cooling chamber surrounding a water-gas generator as a steam-raising boiler. It is difficult to construct such a boiler to withstand high pressures, and also difficult to remove scale from its surface. The present invention uses the cooling chamber for circulating the hot water produced therein, through a closed system in which the heat recovered is transferred to the water being fed to the usual high-pressure boiler, which recovers waste heat from the "blow gases."

S. PEXTON.

Means for detecting the presence of methane or other hydrocarbon gases. J. SEJVL, R. WINKLER, and M. PALKON (E.P. 238,531, 27.7.25. Conv., 15.8.24).—Two spirally coiled filaments, one of tungsten, enclosed in a hermetically-sealed bulb, and the other of some metal which catalyses the combustion of hydrocarbons, and surrounded by a shade through which a current of the gas to be tested is drawn, are connected in series or in parallel with an electric battery. In the centre of each filament is a thermopile, the two being connected in opposition through a galvanometer the null point of which is determined by introducing the apparatus into an atmosphere devoid of hydrocarbons. When the apparatus is brought into the presence of combustible hydrocarbon vapours the active filament increases in

temperature and glows more intensely than the standard filament. Also the thermopile circuit is disturbed to an extent dependent upon the proportion of hydrocarbon vapour present. The galvanometer in the thermopile circuit can be calibrated so as to make the instrument quantitative. S. Pexton.

Recovery of benzol from gases. O. Heitmann (G.P. 431,923, 3.7.21).—The gas is washed with oil of b.p. 180—220° derived from crude benzol, and the benzol absorbed is driven off in stills operating without free steam. The process is characterised by extreme simplicity. W. T. K. Braunholtz.

Distillation of [benzol] wash oil. Bergbau A.-G. Lothringen (G.P. 432,378, 1.3.24).—In order to prevent thickening of the wash oil, the benzol is distilled from it by means of steam which is free from oxygen and carbon dioxide. The steam may be generated from the aqueous condensate in an evaporator placed between the still and the condenser for benzol and steam.

W. T. K. Braunholtz.

Breaking of [petroleum] oil-water emulsions. J. C. Walker, Assr. to Doherty Research Co. (U.S.P. 1,597,700, 31.8.26. Appl., 5.12.21).—The emulsion is treated with a mixture of formaldehyde and steam.

W. N. Hoyte.

Cracking of [hydrocarbon] oils. H. L. Doherty, Assr. to Doherty Research Co. (U.S.P. 1,597,674, 31.8.26. Appl., 22.11.22).—The oil is heated in a pipe still, and the vapours and oil issuing therefrom are passed under pressure and counter-current to one another through a cracking chamber. Pressure on the oil is lowered on entering the evaporator. Residue vapours are condensed by heat-interchange with untreated oil, and residue oil is returned to the cracking chamber. W. N. Hoyte.

Still for cracking [petroleum] oils. D. Pyzel, Assr. to Simplex Refining Co. (U.S.P. 1,597,821, 31.8.26. Appl., 25.6.24).—The oil passes through heating coils to a reaction chamber, and vapours evolved therefrom pass up a dephlegmator. The runback from the dephlegmator is passed again through the heating coils at a rate which varies with the amount of condensate from the dephlegmator. W. N. Hoyte.

Cracking of [hydrocarbon] oils. E. C. Herthel, Assr. to Sinclair Refining Co. (U.S.P. 1,598,136, 31.8.26. Appl., 12.7.22).—Oil is heated in a large still under pressure to cracking temperature until the deposition of pitch begins, fresh stock is then fed in to keep the pitch in solution, and towards the end of the run pitch-laden oil is withdrawn from the still at such a rate that pitch is not deposited. W. N. Hoyte.

Cracking of [hydrocarbon] oils. G. Egloff and H. P. Benner, Assrs. to Universal Oil Products Co. (U.S.P. 1,598,368, 31.8.26. Appl., 23.3.21. Renewed 14.1.26).—The oil is passed through heating coils to a large elongated horizontal expansion chamber, which is fitted with several residuum draw-offs connected to a common header. Fluid under pressure is forced through the header to withdraw residuum quickly from the expansion chamber. W. N. Hoyte.

Lubricating compounds. G. Wisner (E.P. 244,086, 1.12.25. Conv., 5.12.24).—The lubricating qualities of mineral oils are improved by adding 0·5% to 5% of a saturated fatty acid of high molecular volume and containing at least 16 carbon atoms, the best results being obtained when the oil is nearly saturated with respect to the acid; the resistance of the film may be further increased by adding a triglyceride. W. N. Hoyte.

Lubricating compounds. G. Wisner (E.P. 247,520, 1.12.25. Conv., 10.2.25. Addn. to E.P. 244,086). Sulphur is added to the mineral oil in quantities varying from 0·5% to 5%, and fatty acids and triglycerides are then added as set forth in E.P. 244,086 (cf. preceding abstract). W. N. Hoyte.

[Acetylene or oxygen] gas generators. C. Choffel and A. P. Jacquelin (E.P. 243,369, 19.11.25. Conv., 20.11.24).

Gas burners. G. Helps (E.P. 258,632, 17.4.25).

Liquid fuel burners. H. Knudsen (E.P. 258,657, 23.6.25).

III.—TAR AND TAR PRODUCTS.

Determination of anthracene by the Rütgers method. J. Sielisch (Z. angew. Chem., 1926, **39**, 1248—1249).—The method is based on the oxidation of anthracene to anthraquinone, solution of the crude anthraquinone in sodium bisulphite solutions and filtration from impurities, followed by re-oxidation of the anthraquinol, the anthraquinone thus precipitated being finally weighed. The procedure is as follows:—To 1 g. of the sample, dissolved in 45 c.c. of boiling acetic acid, a solution of 15 g. of chromic acid in 10 c.c. of acetic acid and 10 c.c. of water is added at the rate of 1 c.c. per min. until the green solution becomes definitely brown. After boiling for a further ½ hr., the cooled contents are diluted with 400 c.c. of ice-cold water and after ¼ hr. filtered through a glazed filter. The precipitate is washed with ice-cold water then with (at most) 200 c.c. of 1% hot sodium hydroxide, It is then transferred to a 200 c.c. Erlenmeyer flask, and warmed with 15 c.c. of filtered reducing agent (10% sodium hydroxide containing 10% of sodium bisulphite) for a few minutes at 60—80°. The red solution is filtered into a warmed suction flask through filter paper in a Gooch crucible, which has previously been filled with reducing solution diluted 10 times. The crucible is not allowed to run dry throughout the filtration. The residue is submitted to a second reduction with 5 c.c. of reducing agent. The solution is filtered as before and the warm filtrates are oxidised to complete decolorisation with dust-free air or preferably with 5 c.c. of concentrated hydrogen peroxide. The precipitated anthraquinone is filtered through a porcelain filter dish (7 cm. diam.), washed with hot water till neutral, well drained, and dried at 100°. The determination is complete in 3—3½ hrs. instead of a day as in the modified Höchst test (B., 1926, 734) and the experimental losses do not exceed 1% (2% in the Höchst). R. Brightman.

Principles of the determination of anthracene by the Rütgers method. J. Sielisch and P. Köppen-Kastrop (Z. angew. Chem., 1926, **39**, 1249—1253).—A detailed analysis of the procedure in the Rütgers method (cf. preceding abstract) in comparison with the Höchst test. R. Brightman.

Modification of the Höchst method for the determination of anthracene. F. JACOBSOHN (Brennstoff-Chem., 1926, **7**, 311—313).—See B., 1926, 734.

Bromometric determination of the cresols. P. W. DANCKWORTT and G. SIEBLER (Arch. Pharm., 1926, **264**, 439—447).—The official (German) method of analysing liquor cresoli is unsatisfactory. Good results for water, cresols, neutral oils, and soap are obtained by Arnold and Mentzel's procedure (Apoth.-Ztg., 1903, [16]), whilst the following method is applicable when the cresols are to be more exactly determined (cf. also Ulrich and Kather, B, 1926, 306). All the cresols take up three atomic proportions of bromine, but *m*-cresol is brominated much more rapidly than its isomerides. To prevent loss of bromine, a stoppered conical flask is used; the potassium iodide solution (10 c.c. of a 20% solution) is placed in a small sturdy container (*e.g.*, a small bottle) standing inside the flask; and the stopper of the flask is sealed with a little potassium iodide solution. Into the flask are run successively 30 c.c. of 0·1*N*-potassium bromate solution, 25 c.c. of the solution to be analysed (*e.g.*, one containing about 1·5 g. of cresol per litre), 30 c.c. of dilute sulphuric acid (1 part to 5 parts of water), and, after careful mixing, 10 c.c. of 25% potassium bromide solution. The flask is quickly stoppered, and the contents are mixed by swirling, care being taken not to upset the potassium iodide solution. After an appropriate time has elapsed (¼ hr. for *m*-cresol; 24 hours for a mixture; up to 72 hours for *o*- or *p*-cresol) the potassium iodide is upset, and the mixture is titrated with thiosulphate. "Lysol" is acidified and steam-distilled, the distillate being analysed as described. In analysing creolin preparations the neutral oils must be removed from the distillate, either by filtration or a suitable extraction process. W. A. SILVESTER.

PATENT.

Prepared tar for road-making etc. L. H. SENSICLE (E.P. 257,560, 21.5.25).—Prepared tar suitable for road-making can be produced without distillation from a by-product plant by dehydrating a tar or mixture of tars from which part of the condensate rich in naphthalene and phenols has been kept out. In a direct recovery process plant, practically the whole of the output can be converted into prepared tar by collecting separately the condensate from the serpentine coolers and multitubular coolers, removing part of the naphthalene and phenols therefrom, and then mixing the product with the remainder of the tar in the proportions required to produce a tar with the necessary viscosity, distillation range, etc. to comply with the specifications. This prepared tar has the advantage of a low naphthalene and phenol content. If desired the composition can be adjusted by the admixture of tar distillate fractions. B. W. CLARKE.

IV.—DYESTUFFS AND INTERMEDIATES.

New synthesis of phenylrosinduline. A. WAHL and R. LANTZ (Chim. et Ind., 1926, **16**, 355—366).—A *résumé* of work previously published (cf. A. 1923, i, 209; 1926, 296, 530, 617). J. S. H. DAVIES.

Determination of dyes. A. NOLL (Papier-Fabr., 1926, **24**, 593—595; cf. *ibid.*, 1924, **22**, 361).—Methods are given for the determination of auramines and of the sulphuric esters of *leuco*indigoid dyes in the commercial products such as Auramines O and G and Indigosols O and O4B. For the auramines the dye is hydrolysed by boiling dilute hydrochloric acid to the corresponding benzophenone derivative, which is weighed. The soluble indigo derivatives are converted, by hydrolysis and oxidation with dilute sulphuric acid and sodium nitrite, into the corresponding indigos, which are weighed. A. DAVIDSON.

PATENTS.

Anthraquinone-2-glycine-3-carboxylic acid. GES. F. CHEM. IND. IN BASEL (Swiss P. 109,067, 30.11.23. Addn. to 103,217).—2-Chloroanthraquinone-3-carboxylic acid is heated, for example, with glycine (1 mol.), magnesium oxide, water, and copper powder for a long time at 120—130°. The product is acidified, and the precipitate filtered off and purified by dissolving in sodium carbonate solution and reprecipitating. *Anthraquinone-2-glycine-3-carboxylic acid* is an orange powder, its solution in sulphuric acid being deep orange, in alkalis red, in alkaline sodium hyposulphite wine-red. It may be used as an intermediate for dyes. A. DAVIDSON.

Manufacture of 2-mercaptoanthracene-3-carboxylic acid and anthracene-2-thioglycol-3-carboxylic acid. SOC. CHEM. IND. IN BASLE (Swiss P. 110,414 and 110,415, 22.7.24. Addns. to 103,647).—2-Mercaptoanthraquinone-3-carboxylic acid is reduced, *e.g.*, by heating with aqueous ammonia and zinc dust for 4—5 hrs., and the product condensed with chloroacetic acid. 2-*Mercaptoanthracene-3-carboxylic acid* on warming with chloroacetic acid (1 mol.) and alcoholic potash yields the anthracene-2-thioglycol-3-carboxylic acid described in the chief patent. A. DAVIDSON.

Manufacture of anthraquinone-thioxanthones and -acridones. SOC. CHEM. IND. IN BASLE (Swiss P. 110,749, 111,500, and 111,501, 16.4.24).—A 1-chloro-4-acylaminoanthraquinone or 1-chloro-4-aminoanthraquinone (1 mol.) is condensed with 5-chloro-1-mercaptobenzene-2-carboxylic acid or 5-chloroaniline-2-carboxylic acid (1 mol.), the *N*-acyl group in the products eliminated where necessary, and ring closure to thioxanthones or acridones brought about by condensing agents such as chlorosulphonic acid. For example, 1-chloro-4-carbethoxyaminoanthraquinone is heated at 120—130° with 5-chloro-1-mercaptobenzene-2-carboxylic acid, potassium or sodium carbonate, and amyl alcohol, the alcohol distilled off, the residue extracted with dilute sodium hydroxide, and the alkaline solution acidified. The violet-brown flocks of 4-*carbethoxyaminoanthraquinonyl-1-mercapto-5′-chlorobenzene-2′-carboxylic acid* are hydrolysed by 90% sulphuric acid at 15—20° to the free amino-compound. On adding this to chlorosulphonic acid and keeping the solution at 40° till a sample, diluted with ice water, gives a pure blue precipitate, *anthraquinone-4′-chloro-4-amino-2 : 1-thioxanthone* is formed. It gives a reddish-orange solution in sulphuric acid and a violet vat with alkaline sodium hyposulphite. 4-*Aminoanthraquinonyl - 1 - mercapto - 5′ - chlorobenzene - 2′ - carboxylic*

acid, violet-brown, can also be obtained by condensation of 5-chloro-1-mercaptobenzene-2-carboxylic acid with 4-chloro-1-aminoanthraquinone in presence of aqueous sodium hydroxide and alcohol at 80°. On heating 1-chloro-4-carbethoxyaminoanthraquinone with 5-chloroaniline-2-carboxylic acid, potassium carbonate, a little cuprous chloride and amyl alcohol for a long time under reflux there is formed 4-*carbethoxyamino*-1 : 5′ : 2′-*chlorocarboxyphenylaminoanthraquinone*. This is hydrolysed by 85% sulphuric acid at 15—20° to 4-*amino*-1 : 5′ : 2′-*chlorocarboxyphenylaminoanthraquinone*. On heating the latter with chlorosulphonic acid at 15° till the blue solution becomes red, ring-closure takes place with formation of *anthraquinone*-4′-*chloro*-4-*amino*-2:1-*acridone*, blue, giving a violet vat with alkaline sodium hyposulphite. The products are used for the preparation of dyes. A. Davidson.

Manufacture of 2 : 3-anthraquinonylindoxyl. Soc. Chem. Ind. in Basle (Swiss P. 110,416, 30.11.23. Addn. to 103,648).—Anthraquinone-2-glycine-3-carboxylic acid is heated under reflux for some time with anhydrous sodium acetate and acetic anhydride. The brownish-yellow crystals which separate are washed with acetic acid, and then with water. *Anthraquinonyl*-2 : 3-*indoxyl* dissolves in cold concentrated sulphuric acid to an orange-red solution from which water precipitates greenish-yellow flocks. With alkaline sodium hyposulphite it gives at first a brown, then an olive-green vat. It serves as an intermediate for dyes. A. Davidson.

Manufacture of anthracene-2 : 1-thioindoxyl. Soc. Chem. Ind. in Basle (Swiss P. 111,997, 14.1.25).—Anthracene-2-thioglycollic acid, obtained either by condensation of 2-mercaptoanthracene with chloroacetic acid or by reduction of anthraquinone-2-thioglycollic acid, is treated with condensing agents. The *anthracene*-2-*thioglycollic acid*, yellow, m.p. 105°, is converted by phosphorus pentachloride into anthracene-2-thioglycollyl chloride. This is dissolved in chlorobenzene, treated with aluminium chloride, and warmed for several hours until evolution of hydrogen chloride ceases. The reaction mixture is added to ice and hydrochloric acid, the chlorobenzene driven off by steam, and the residue extracted with dilute sodium hydroxide to obtain *anthracene*-2 : 1-*thioindoxyl*, yellow, m.p. 172°. It finds application as an intermediate for dyes. A. Davidson.

Anthraquinoneacridine dye. Soc. Chem. Ind. in Basle (Swiss P. 111,502, 13.5.24. Addn. to 97,059).—8-Amino-2 : 1-anthraquinoneacridone (less than 3 mols.) is mixed with nitrobenzene, cyanuric chloride (1 mol.) is added, and the mixture warmed gradually to 190—200° and stirred for several hours at this temperature. The condensation product, when washed with nitrobenzene and alcohol, is obtained as a dark violet powder, which dyes cotton from the vat in reddish-violet shades fast to light, washing, and chlorine. A. Davidson.

Aromatic amines (G.P. 421,151).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of the dry weight of wool. S. G. Barker and J. J. Hedges (J. Text. Inst., 1926, **17**, T 453—456).—A comparison of the actual moisture content of wool with the commercial regain as determined by an ordinary conditioning oven and by the Bradford Conditioning House has shown that the latter figure is only about 0·5% less than when specially dried air is used in the drying vessel, and is therefore sufficient for practical purposes. The common ovens, however, give results about 1% too low, and frequently they may be found in works near steam-using plant or other places where the air is unduly moist. J. C. Withers.

Gel structure of the wool fibre. J. B. Speakman (J. Text. Inst., 1926, **17**, T 457—471).—In this and a subsequent paper (*ibid.*, 472), it is shown that the wool fibre has a true yield point and, after extension, remains permanently more extensible under low loads. Such observations are held to necessitate the replacement of the theory that wool has a two-phase gel structure by a four-phase theory. It is argued that the wool fibre consists of two gels arranged, as it were, in parallel. The first may be called the petrified gel, and comprises an elastic cell wall enclosing a fibrillar structure which is not in physical equilibrium with a viscous phase. The second, which fills the interstices of the petrified gel, is gelatinous and capable of reversible solution in, and deposition from, water. Gelation occurs at 0° in water, and at ordinary temperatures in unsaturated air and, under such conditions, the medium possesses the usual two-phase structure of gels. In reality, therefore, the wool fibre consists of four phases, but for convenience these may be reduced to three, viz. (1) an elastic cell wall enclosing (2) a fibrillar structure which is not in physical equilibrium with (3) a viscous, gelatinous medium included in its interstices. The experiments described deal with breaking loads, extensions at break, and breaking stresses, and include measurements of the effect on these factors of diazotisation, formaldehyde, benzoquinone, boiling water, and chlorine water. J. C. Withers.

Extension of wool fibres under constant stress.—J. B. Speakman (J. Text. Inst., 1926, **17**, T 472—481).

Viscose. E. Berl and J. Bitter (Cellulosechem., 1926, **7**, 137—145).—The xanthate reaction is a specific reaction of the alkali alkoxide group, and depends intimately on the alcohol–alkoxide equilibrium; in the case of polyhydric alcohols considerable resistance is experienced. Primary monohydric alcohols, like ethyl alcohol, react easily and completely, glycol forms a xanthate slowly with simultaneous formation of by-products, glycerol under ordinary conditions does not form a xanthate at all. Methylation alters these relationships so that glycol monomethyl ether and glycerol dimethyl ether react easily, like ethyl alcohol, whilst glycerol monomethyl ether reacts like glycol. Surface phenomena also play a part, so that in the presence of active charcoal even glycerol may form a xanthate. Viscoses can be prepared from cellulose monoalkyl ethers, but the dialkyl ethers cannot be brought into reaction; hence the presence of two free hydroxyl groups in the case of cellulose is essential to xanthation. The alkali first produces the sodium alkoxide, for which purpose in order to establish a favourable equilibrium it must be concentrated, and then hydrates closed anhydride linkages, thus altering the physical structure

of the cellulose, increasing its internal surface development and setting up the initial stages of dispersion. The absence of stoicheiometrical proportions between the reacting carbon disulphide and cellulose suggests that while xanthate groups are formed as the result of the alkali alkoxide equilibrium the most important feature of the reaction is the breakdown of the cellulose substance into a dispersed condition. Whereas xanthation occurs in the presence of a restricted quantity of water, hydrolysis proceeds in the presence of an excess of water. During the ripening process, the ultramicroscopic, dispersed particles show a progressive slowing down of their Brownian motion with ultimate cessation; this is accompanied by a noted decrease in the number of particles and increase in their size until incipient coagulation is indicated by a slightly coherent structure which finally develops to a honey-comb gel. The hydrolysis of the xanthate in dilute solution is characterised by the formation of free cellulose xanthic acid and caustic soda in equilibrium. The free xanthic acid dissociates into dispersed hydrated cellulose and carbon disulphide which reacts with the sodium hydroxide. The equilibrium is thus upset and the hydrolysis proceeds further, while the accumulation of dispersed free cellulose induces re-aggregation of the particles. J. F. BRIGGS.

Viscose. E. BERL and A. LANGE (Cellulosechem., 1926, **7**, 145—147).—Viscose prepared from cotton linters has a higher viscosity than that from wood cellulose, under identical conditions, corresponding with the higher micellar molecular weight of the cotton cellulose. Both types of viscose during the ripening process undergo an initial fall in viscosity which is attributed to the degrading action of the sodium hydroxide. After this initial fall there is a continuous rise in viscosity. If the viscose be precipitated at the different stages of ripening, either by neutral or acid coagulating agents, converted into hydrated cellulose, and if this regenerated cellulose be nitrated under standard conditions, the viscosity of the nitrocellulose solutions in acetone may be taken as a measure of the integrity of the cellulose molecule in the viscose at that stage of ripening. The results showed that the nitrocelluloses from the cotton viscose had consistently higher viscosities than those from wood-pulp viscose. The nitrocelluloses from freshly prepared viscose had higher viscosities than those which had ripened for a few hours. On the other hand, the subsequent stage during which the viscosity of the viscose continuously increases during ripening is not correspondingly reflected in the viscosity of the nitrocellulose prepared from the regenerated cellulose. After the initial rapid fall in the viscosity of the nitrocellulose, this remains constant during further ripening of the viscose. J. F. BRIGGS.

Experimental method for investigating thermal properties of cotton fabrics. J. GREGORY (J. Text. Inst., 1926, **17**, T 553—566).

Changes in strength and elongation of artificial silk caused by moisture. Y. KAMI (Cellulose Ind., Tokyo, 1926, **2**, 25).—The elongation of artificial silk generally increases 10—65% on wetting, the average being 40%, but the elongation of some kinds diminishes. The tensile strength diminishes by 50—70%. These changes are independent of the duration of moistening, and the original properties of the silk are almost recovered on drying. The cross-section of the dried thread is elliptical or rectangular, but when wetted it becomes almost circular and 2—2·5 times larger in area. A. GEAKE.

Viscose. V. G. KITA and R. TOMIHISA (Cellulose Ind., Tokyo, 1926, **2**, 26—28).—During the ripening of viscose, xanthate is formed by the action of carbon disulphide on alkali cellulose, and sodium carbonate and thiocarbonate are formed from carbon disulphide and free sodium hydroxide. The first reaction is the faster, but the xanthate again decomposes when the concentration of carbon disulphide and sodium hydroxide is reduced by the second reaction. Eventually, all the free alkali is exhausted, and the viscose is coagulated by the accumulation of salts. Rise of temperature favours the formation of carbonate and thiocarbonate because the xanthate decomposes more rapidly. During spontaneous coagulation all free alkali disappears. A. GEAKE.

Viscose. VI. G. KITA, K. AZAMI, and R. TOMIHISA (Cellulose Ind., Tokyo, 1926, **2**, 28—30; cf. preceding abstract).—Increasing the amount of carbon disulphide from 100% to 150% of the weight of cellulose increases the viscosity, but does not affect the proportion of combined alkali (about 0·65 mol.). Increasing the amount of alkali diminishes the viscosity and increases the proportion combining; the ratio of carbonate to thiocarbonate is also increased. If xanthate is diluted with 5% sodium carbonate solution instead of with water, the combination is stronger, and the viscosity is diminished. Addition of sodium hydroxide, which has been shaken with carbon disulphide until no free alkali remains, results in less combined alkali and a higher viscosity than when the sodium hydroxide and carbon disulphide are added without previous combination. Combination after a definite time of ripening is increased by the addition of sodium hydroxide to a maximum, which is probably determined by the exhaustion of the carbon disulphide; combination is then further increased by the addition of carbon disulphide. A. GEAKE.

Cellulose esters. G. KITA, I. SAKRADA, and T. NAKASHIMA (Cellulose Ind., Tokyo, 1926, **2**, 30—32).—A cellulose ester containing 0·29 mol. of fatty acid may be further esterified after treatment with sodium hydroxide. After 14 esterifications the proportion of fatty acid reaches a maximum of 2·11 mols. About half of this ester is soluble in benzene, the soluble part containing 2·32, and the insoluble 2·00 mols. of fatty acid. Unchanged cellulose may be extracted with cuprammonium hydroxide solution, or by the viscose process, from esters containing 1 mol. of fatty acid to 2, 3, or 4 $C_6H_{10}O_5$ groups, and the composition of the undissolved residue tends to approach 1 mol. of fatty acid to one $C_6H_{10}O_5$ group. It is concluded that in cuprammonium hydroxide solution cellulose is dispersed into $C_6H_{10}O_5$ units. Ripened xanthate does not combine with fatty acid chlorides after purification with acetic acid and salt solution, but when precipitated with alcohol a sulphur-free ester may be obtained. A. GEAKE.

Polysaccharides. XXXV. Enzymic degradation of artificial silk and natural cellulose. (P. Karrer and P. Schubert (Helv. Chim. Acta, 1926, 9, 893—905).—Different varieties of cellulose and artificial silks show widely differing reactivity towards cellulase, previous treatment by alkali greatly aiding reaction. Tables are given showing rate of destruction for many commercial preparations, with diagrams showing the magnified cellulose particles, from which it appears that increase of surface favours enzyme action. Artificial silk which is easily dyed is easily attacked by cellulase. The velocity of decomposition of filter-paper by cellulase was determined, but no velocity constant could be found.

E. W. Wignall.

Semi-chemical pulping process. J. D. Rue, S. D. Wells, F. G. Rawling, and J. A. Staidl (Pulp and Paper Mag., 1926, **24**, 1163—1167).—A modified digestion process is described which is particularly applicable to hard woods such as aspen, birch, and the gums. High yields (70—85%) are obtained, and the pulp is suitable for medium- and low-grade printing papers and certain classes of board. The process involves impregnation of the chips under pressure, mild digestion with chemicals which are practically neutral, and are capable of maintaining approximate neutrality during the digestion operation, and reduction of the softened chips by mechanical means. A suitable digestion liquor consists of a mixture of sodium sulphite and sodium bicarbonate, conveniently prepared by absorbing sulphur dioxide in soda ash solution; the ratio of sodium bicarbonate to sodium sulphite may vary within wide limits, depending on the character of the wood and the quality of pulp required: the total quantity of chemicals need not exceed 15 lb. per 100 lb. of wood. The chips, after steaming for at least $\frac{1}{2}$ hr. at atmospheric pressure, are impregnated with the cooking liquor under a pressure of 100 lb./sq. in., preferably at 120—125°, for 1—$1\frac{1}{2}$ hrs. After removal of the excess of liquor the temperature of the cook is quickly raised to 140—160° and held there for 1—6 hrs. The digested chips are washed and finally converted into pulp by passage through a rod mill. Semi-chemical pulp from aspen and birch may satisfactorily replace over 50% of a standard newsprint furnish, or may be used with ground wood alone; in the latter case, however, the paper, though strong when dry, is relatively weak in the wet state and somewhat difficult to run on the machine. In the manufacture of boards, less care is necessary in the selection, preparation, and cooking of the wood, and dark woods, such as chestnut, elm, etc., may be used. Chestnut chips (after extraction of tannin) have been treated by this process on a commercial scale with satisfactory results. There appears to be no advantage in treating coniferous woods by a semi-chemical process since the resulting pulp lacks strength and felting qualities.

D. J. Norman.

Micro-organisms concerned in the decomposition of cellulose in the soil. Waksman and Skinner.—See XVI.

Patents.

Treating textile material. R. A. Phair, Assr. to H. Kohnstamm & Co., Inc. (U.S.P. 1,598,305, 31.8.26. Appl., 16.7.21).—In the process of boiling out fats and waxes from vegetable fibres and textiles prior to bleaching, magnesium oleate is added to the alkaline water to the extent of 10% of the alkali present.

R. B. Clarke.

[Electrolytic] manufacture of cellulose. T. A. Hill, Assr. to Taylor Laboratories, Inc. (U.S.P. 1,590,595, 29.6.26. Appl., 26.5.20. Renewed 17.6.24).—A cuprammonium solution of cellulose is fed continuously to an electrolytic cell having a platinum or platinum-plated belt as anode and a suitable metallic cathode. The belt travels in the same direction as the solution, and cellulose is deposited on each side of the belt in increasing thickness, which can be regulated by suitable means. The films of cellulose are removed by means of brushes and passed through a tank containing dilute sulphuric acid, and finally washed and wound.

R. B. Clarke.

[Electrolytic] manufacture of cellulose products. E. Taylor, Assr. to Taylor Laboratories, Inc. (U.S.P. 1,590,603, 29.6.26. Appl., 17.6.24).—In the process of U.S.P. 1,590,595 (cf. preceding abstract) the platinum or platinum-plated belt is replaced by a copper belt.

R. B. Clarke.

Manufacture of artificial filaments [cellulose acetate]. G. B. Ellis. From Soc. Fabr. de la Soie "Rhodiaseta" (E.P. 248,696, 27.5.25).—In the process of dry-spinning solutions of cellulose derivatives in volatile solvents, the cross-sections of the filaments are controlled, the flat type giving scintillating effects being avoided and the rounded or star-shaped type being produced, by ensuring at the spinning apertures an input of heat, maintaining at the spinning dies and in the immediate neighbourhood a temperature regulated and varied according to the demand of the other spinning conditions, independently of the temperature of the remainder of the cell.

J. F. Briggs.

Subjecting wood and similar materials to a preliminary treatment for the manufacture of paper board etc. H. Bräunlich (U.S.P. 1,597,717, 31.8.26. Appl., 29.5.25. Conv., 30.5.24).—The process of steaming wood for the manufacture of paper boards is divided into four separate stages. The material is first subjected to a slow preparatory heating up to a steam pressure of 2—4 atm. according to quality; in the second step this pressure is kept approximately constant for 2—4 hrs., and in the third step it is gradually reduced to atmospheric pressure within 3—8 hrs. by regulating the steam supply. The fourth step consists in admitting water to the boiler, with or without the admixture of chemicals, and saturating the material therewith for 4—5 hrs.

J. F. Briggs.

Collapsible reels for winding filaments, yarns, threads, silks, etc. Brysilka, Ltd., and F. W. Schubert (E.P. 258,377, 2.7.25).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Patents.

Machines for dyeing and similarly treating fabrics. F. Riley (E.P. 258,736, 28.9.25).

Yarn beams on which yarns are to be wound for fluid treatment. J., T., and J. Brandwood (E.P. 258,309, 15.5.25).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Neutralisation of ammonium sulphate. A. E. THAU (Gas- u. Wasserfach, 1926, **69**, 832—834).—A *résumé* of Bateman's work (B., 1926, 404).
R. A. A. TAYLOR.

Manufacture of sodium dichromate. L. L. POPOV (J. Chem. Ind. [Russia], 1925, **2**, 465—474; Chem. Abstr., 1926, **20**, 3063).—Rich chromium ores are more advantageous than poor ores, as regards both speed of oxidation and yield of dichromate; poor ores must be finely pulverised. The amount of lime must be 80% of the weight of ore if the latter contains 30—40% Cr_2O_3, 90% for 40—50%, or 120—130% for a content greater than 50%. These quantities must be increased if the temperature of operation is above 1100°. The theoretical quantity of sodium carbonate is employed. Rich ores are calcined for 1½ hrs. at 1100°; poor ores for a longer time at 1200°. A. A. ELDRIDGE.

Synthesis of galena crystals. W. OGAWA, C. NEMOTO, and S. KANEKO (Res. Electrochem. Lab., Japan, 1926, [173], 1—59; Chem. Abstr., 1926, **20**, 2768).—Galena crystals, suitable for use as radio detectors, were obtained by fusion of (i) lead sulphide 92·5, silver sulphide 7·5, and sulphur 1·5 parts, or (ii) lead sulphide 99·5, thallous sulphide 0·5, sulphur 1·5 parts.
A. A. ELDRIDGE.

Recovery of sulphur from gases. FISCHER and DILTHEY.—See II.

PATENTS.

Apparatus for hydrating lime. H. MISCAMPBELL (E.P. 257,793, 26.11.25).—The lime, after treatment in a pulveriser situated below ground level, is discharged by a vertical elevator into a bin, and is fed by a reciprocating feeder and a spiral conveyor into a horizontal mixer fitted with rotating paddles. Water is added, and the lime and water are discharged into a hydrator pan rotating upon rollers or ball bearings; in the pan are hung stationary ploughs which stir the mixture during the slaking process. The hydrated lime is discharged through the bottom of the rotating hydrator, and is elevated to a series of horizontal cooling conveyors in which the lime is carried in one direction through one conveyor, and in the opposite direction through another, until it is discharged and elevated to a final pulveriser. The dust and fumes evolved during the mixing and hydration are condensed in a dust collector provided with water-spraying nozzles, and are sent back with the water to the mixer. W. G. CAREY.

Production of pure hydroxides of barium or strontium. RHENANIA VEREIN CHEM. FABR. A.-G., Assees. of B. STUER (G.P. 432,114, 9.8.24).—Barium or strontium oxides or hydroxides containing sulphide, which have been obtained from the sulphur compounds by hydrolysis without the use of ammonia, are crystallised from water in presence of ammonia.
W. G. CAREY.

Production of sodium sulphate from ferrous sulphate and sodium chloride. VEREIN FÜR CHEM. U. METALL. PROD. (G.P. 432,201, 21.10.24).—After heating, the undecomposed sodium chloride is removed by extraction with water at so low a temperature that the smallest amount of sodium sulphate is dissolved, the residue containing sodium sulphate is then dissolved in water at a suitable temperature and the sodium sulphate is separated. W. G. CAREY.

Simultaneous production of potassium phosphate and phosphoric acid. W. H. ROSS, R. M. JONES, and A. L. MEHRING (U.S.P. 1,598,259, 31.8.26. Appl. 4.3.24).—A mixture of phosphate rock, a potash silicate, and a carbonaceous material is heated in a reducing atmosphere at 1300°. The evolved fumes are burnt at the outlet of the furnace and the resulting product is recovered in a Cottrell precipitator.
S. BINNING.

Calcium cyanogen compounds. R. W. POINDEXTER, Assr. to CALIFORNIA CYANIDE Co., INC. (U.S.P. 1,596,119, 17.8.26. Appl., 18.2.26).—Solid cyanides of the alkaline-earth metals are prepared by treating a mixture of the oxide of the metal and a monohydric alcohol, *e.g.*, methyl alcohol, with an excess of hydrocyanic acid. R. B. CLARKE.

Cyanide product [containing combined ammonia]. R. W. POINDEXTER, Assr. to CALIFORNIA CYANIDE Co., INC. (U.S.P. 1,596,120, 17.8.26. Appl., 13.4.26).—The compound $Ca(CN)_2,2NH_3$ is prepared by the interaction of hydrocyanic acid and calcium nitrate tetrahydrate in anhydrous liquid ammonia. The product decomposes in air and is used as an insecticide. R. B. CLARKE.

Production of arsenic trisulphide. I.-G. FARBENIND. A.-G., Assees. of U. DREYER (G.P. 432,358, 16.1.25).—In the production of arsenic trisulphide from arsenious oxide and hydrogen sulphide, the arsenious oxide is suspended in water or in a salt solution, which preferably is warmed. W. G. CAREY.

Manufacture of hydrogen. J. H. BEAUMONT (E.P. 257,434, 14.10.25).—Pure precipitated ferric oxide is reduced to iron by producer gas in an upper vessel which connects through its base with a lower vessel so that the iron passes into the latter by gravity without contact with air. The iron is treated with steam in the lower vessel, giving pure hydrogen, and being oxidised to ferrous oxide, which is then elevated by suitable means to the upper vessel and again reduced. The reduction is carried out between 500° and 550°, since to ensure the production of pyrophoric iron the temperature must not exceed 600°, and the re-oxidation takes place between 500° and 650°, the temperature being kept below 700° to minimise the formation of magnetic iron oxide, which requires more carbon monoxide for its reduction. The temperatures are controlled by external heating.
W. G. CAREY.

Treatment of solids with liquids (E.P. 257,643).—See I.

VIII.—GLASS; CERAMICS.

Effects of thermal treatment on glass as shown by precise viscosimetry. V. H. STOTT, D. TURNER, and H. A. SLOMAN (Proc. Roy. Soc., 1926, **A, 112**, 499—521; cf. B., 1925, 921).—A method of determining the viscosity of molten glass by measuring the

thickness of the film which adheres to a fine iridio-platinum wire withdrawn from it at a known rate is described, the attainable accuracy being $\pm$ 3%. By this means numerous experiments have been made with the glass N.P.L. 15. It is found that the viscosity of this glass at temperatures above 1200° is not altered by cooling the glass to the ordinary temperature and re-heating, unless the glass is kept too long within a certain temperature range below 1200°. In this region the glass is apparently capable of existing in at least two states of differing viscosity. These facts may have some connexion with the known effect of variations in, *e.g.*, the moisture content, heat treatment, or proportion of cullet on the brittleness and mechanical behaviour during working of glasses of the same chemical composition.

R. Cuthill.

Machine-made table glass. W. Schmidt (Keram. Runds., 1926, **34**, 521—523; Chem. Zentr., 1926, **97**, II, 1567).—Ordinary table glass is not adapted to mechanical manufacture, on account of a tendency to devitrification. A glass containing 11% of calcium oxide and 19% of sodium oxide is suitable for the Foucault process but it is not very stable. Stability is conferred by the addition of alumina, and such a glass is " long," very resistant to tension and pressure, tough, and, at the same time, elastic. Alumina can be introduced in the form of an aluminous sand. Koalin is incorporated into the glass with difficulty, alumina more easily, and felspar most easily of all. L. S. Theobald.

Separation of metals from glass melts. L. Springer (Keram. Runds., 1926, **34**, 503—504; Chem. Zentr., 1926, **97**, II, 1567).—Lead is produced on melting lead-glass under reducing conditions. Melts of copper-ruby glass and silver-yellow glass yield copper and tin, and silver respectively. In another case copper and iron separated, the latter probably arising from ferrous oxide. A melt of table glass gave copper and iron either in a compact form or as iron tinsel. A piece of cast iron added to the melt absorbed copper, but was still undissolved after 3 days. A change in shape and loss of weight, however, were observed. L. S. Theobald.

Vitreous silica and vitreous quartz. W. W. Winship (Trans. Amer. Electrochem. Soc., 1926, **50**, 177—188).—Vitreous silica and vitreous quartz exhibit the following properties: indifference to acid reagents, small expansion coefficient, high temperature electrical resistance, low solubility in water, good light transmission over the whole spectrum, low permeability to gases, low dielectric loss, and simple chemical structure. Applications of these products to various chemical, electrical, and mechanical purposes, also to optical apparatus and in the gas industry, are described, all being dependent upon one or more of these properties. F. G. Crosse.

Temperature diffusivities and thermal conductivities of silica and fireclay refractories. A. T. Green (Gas World, 1926, **85**, Coking Sect., 112—114).—The diffusivities of silica products at lower (700°) and at higher temperatures (1200°) are, in general, higher than those of fireclay products, although individual products may vary from this generalisation. The lower values of the apparent specific gravity of well-fired silica bricks tend to give lower values for the thermal conductivities when compared with fireclay materials. Well-fired silica bricks of good quality, in some cases, are better conductors of heat than the average fireclay brick at 800°, whilst many silica and fire-bricks have approximately equal conductivities at this temperature. At higher temperatures (1200°) silica bricks are the better conductors, except in the case of those which have been inadequately fired. The rate of increase of diffusivity and conductivity with temperature is generally greater with silica products. The effect of porosity and texture on conductivity is briefly discussed. L. S. Theobald.

Effect of industrial usage on the thermal conductivity of a semi-silica material used in a coke-oven wall. A. T. Green (Gas World, 1926, **85**, Coking Sect., 111—112).—Diffusivity and thermal conductivity data, determined by the method previously described (Trans. Cer. Soc., 1922, **21**, 394; cf. B., 1922, 547 a), are reported for semi-silica material which had been in use for 18 years and for a sample of the original, unused material. The used material shows higher values for thermal conductivity over the range 500—1300°, and this is ascribed to alterations in the constitution of the material and in the nature of its porosity. L. S. Theobald.

Thermal expansion of some fused oxides used as refractories. G. E. Merritt (Trans. Amer. Electrochem. Soc., 1926, **50**, 283—291).—The thermal expansions of silica, zirconia, thoria, the refractories made of magnesia and alumina, and of a mixture of equimolecular proportions of thoria and zirconia, between the temperatures 25° and 1000°, were measured. The results are exhibited graphically and compared. The introduction of 3·5% SiO_2 + 0·35% TiO_2 to pure zirconia appreciably raises the thermal expansion. From the form of the curves, and other evidence, it is concluded that combination takes place when the equimolecular mixture of thoria and zirconia is fused at about 3500±500°, giving a crystalline thorium zirconate. The average coefficients of expansion of these oxides from room temperature to 800° are tabulated.

F. G. Crosse.

Application of recrystallised silicon carbide. F. A. J. Fitzgerald (Trans. Amer. Electrochem. Soc., 1926, **50**, 279—282).—Recrystallised silicon carbide, made by mixing silicon carbide with glue as a binder and heating to about 1800°, has been satisfactorily used as a refractory material in furnaces for firing porcelain.

F. G. Crosse.

Refractories for melting metals. Jordan and others.—See X.

Thermal insulation of electric furnaces. Hartmann and Westmont.—See XI.

Refractories for induction furnaces. Unger. —See XI.

Patents.

Manufacture of refractory substances from high-grade oxide of zirconium. Deutsche Gasglühlicht-Auerges. m.b.H. (E.P. 245,101, 16.12.25. Conv., 24.12.24).—Refractory substances capable of resisting temperatures exceeding 2000° are manufactured by mixing up to 5% of the oxide of an alkaline earth, *e.g.*, magnesium oxide, with high-grade zirconium oxide,

pressing the wet mixture, and sintering at 1300° preliminary to finishing at a higher temperature. More porous articles can be prepared by dispensing with the preliminary sintering. B. W. CLARKE.

Manufacture of glass, sand, or flint paper, emery cloth, and like abrasives. C. A. KLEIN and R. S. BROWN (E.P. 258,412, 26.8.25).—The base of sheet abrasive material is first waterproofed by passing it through a bath of linseed oil and wax kept at 100—170°. The abrasive is fixed to this base by means of an adhesive, similarly waterproofed with a drying oil and a gum or synthetic resin. The material thus prepared is unaffected by moisture. F. G. CROSSE.

Producing moulded articles from refuse or its constituents. A. GROTE (E.P. 257,854, 14.4.26).—Calcium carbonate and silicious materials are added during the fusing of refuse etc. so that the silica–lime ratio is from 2 : 1 to 4 : 1, the slag produced still remaining plastic when cooled to 900°. In this condition it can be moulded under pressure to form homogeneous moulded articles which are subsequently subjected to a tempering process, the final product resembling basalt in character. B. W. CLARKE.

Kilns and other heat treatment furnaces. MORGAN CRUCIBLE CO., and C. W. SPEIRS (E.P. 257,472, 18.12.25).—Electrically heated tunnel kilns are provided with trucks for supporting the ware, furnished with tables arranged to rotate as the truck is traversed through the kiln, whereby the articles are subjected to an approximately uniform heat treatment. B. W. CLARKE.

Oil- or gas-fired [pottery] kilns. W. S. MURRAY (E.P. 258,318, 4.6.25).—In kilns used for firing pottery, a heat economy is effected by having the kilns arranged in tandem or series each communicating with the next by a passage at the bottom, and the last leading into a shaft. Burners are so arranged inside each kiln that the products of combustion, after heating the first kiln, pass on to the next, which thus becomes partly warmed. F. G. CROSSE.

Manufacture of sheet glass. E. C. R. MARKS. From E. DANNER (E.P. 258,694, 6.7.25).

IX.—BUILDING MATERIALS.

Mortar sands. H. BURCHARTZ (Zement, 1926, [8]; Mitt. Materialprüf., 1926, [3], 60—62).—Sand from the Rhine will vary according to its source in particle size and density (bulk weight) with corresponding variations in strength of the mortar produced. It should not be used without preliminary tests being carried out. The density (bulk weight) of a sand is not always a measure of its suitability for use as mortar. Although the addition of fine sand to coarse sand, to increase the density, will also increase the strength of the mortar produced, yet a further increase of density obtained by the addition of stone dust will not bring about a corresponding increase in strength of the mortar, owing to the excessive increase in the surface area of the particles which must be covered by the cement. B. W. CLARKE.

Coloration of cement, concrete, and other building materials. C. J. GOODWIN and G. N. WHITE (Chem. Age, 1926, 14, 435).—A solution of a suitable dyestuff or its derivatives is used instead of water for gauging cement or concrete, so that during mixing and setting, oxidation takes place and the original dyestuff is precipitated in the cement. In this way the particles of cement become covered with the particles of dyestuff, and it is possible to obtain suitable shades of colour with the use of 0·1—0·6% of colouring matter reckoned on the dry weight of the concrete. The depth of the tint produced depends only on the ratio of dyestuff to cement, being unaffected by the addition of aggregate. The dyestuff used must be fast to light, moisture, and atmospheric influences, and in the finished state must be completely insoluble in water. B. W. CLARKE.

Analysis of burnt chalk and magnesite after storage in air. A. STETTBACHER (Z. angew. Chem., 1926, 39, 1151—1154).—Drying the stored material in phosphorus pentoxide-sodium desiccators shows that a proportion of the water taken up is not chemically combined. Freshly-ignited pure magnesium oxide after exposure to air for 3—4 days is found to contain 1·8—2·1% of water removed on keeping in the desiccator. Calcium oxide similarly treated and kept for three days lost 0·6—0·7% in weight on keeping in a vacuum desiccator. S. I. LEVY.

Painting of treated wood. M. E. DUNLAP (Ind. Eng. Chem., 1926, 18, 1091).—Paint exposure-tests were carried out on wood panels previously treated with creosote, mixtures of creosote and montan wax, gas oil, pine oil, sugar, borax, zinc chloride, sodium fluoride, mercuric chloride, and two proprietary water-soluble articles, Triolith and Ac-Zol. While the water-soluble preservatives give rise to no apparent detrimental action, discoloration, bleeding, checking, and peeling are observed in the other cases. The strengths of solution and average absorptions of the preservatives by the wood are tabulated. S. S. WOOLF.

PATENTS.

Continuous furnace for preparing molten aluminous cement, etc. G. M. J. DUMAS (E.P. 257,819, 3.2.26).—A continuous furnace for producing aluminous cement or other products having a high melting point consists of a melting chamber of the reverberatory type provided with a water jacket for cooling the walls at the places most exposed to corrosion, and a feeding channel, preferably a vertical furnace of the cupola or water-jacket type, through which only part of the burnt gases is passed while the material to be treated is delivered. The material in the reverberatory furnace moves down a slope and is heated by one or more flame jets under pressure (preferably burning pulverised coal), the pressure in the furnace being kept above atmospheric by a proper adjustment of the draught and of the flames. Openings are provided which serve as tap holes for the molten cement and for the escape of part of the exhaust gases, being made large enough to allow the passage of the whole of the burnt gases without affecting the rate of combustion if the feeding channel becomes choked through partial fusion of the material to be treated. In normal conditions, some of these openings are kept closed by clay stoppers, which are broken when the feeding channel is choked, and are arranged so that

the flames in this event are directed against the partially fused lump of material, thereby freeing the channel and ensuring continuity of operation. B. W. Clarke.

Apparatus for calcining and clinkering with recovery of by-product heat and by-products. R. D. Pike (U.S.P. 1,596,509, 17.8.26. Appl., 21.8.24). —The raw mix for making Portland cement clinker, after being calcined in a neutral atmosphere, is passed to an inclined rotary kiln into which sulphur dioxide is passed from sulphur burners. The kiln is heated to 1370—1540° and a free supply of air is maintained. The kiln gases are then treated for the separation of alkali sulphates as described in U.S.P. 1,594,689 (cf. B., 1926, 879). R. B. Clarke.

Protection of wood from natural discoloration. G. Grau. (G.P. 432,300, 8.5.25).—The wood is coated with a solution of *p*-toluenesulphonamide hydrochloride or one of its soluble salts, and containing an alkali carbonate, so that it is protected from decomposition by metal salts. The solution itself causes no discoloration and offers the discolouring fungi an alkaline medium, in which the growth of the fungus *Ceratomella* and of other moulds causing discoloration of wood is very markedly inhibited. For example, the wood, as soon as possible after cutting, is painted with or steeped in a 0·5—1% solution of *p*-toluenesulphonamide hydrochloride or one of its soluble salts to which is added a 5% solution of sodium carbonate. W. T. K. Braunholtz.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Passivity and corrosion of iron. L. McCulloch (Trans. Amer. Electrochem. Soc., 1926, **50**, 197—205).—Of a number of small particles of electrolytic iron isolated from one another, only a certain proportion will rust in the presence of air, moisture, and sodium chloride. The smaller the particles, the greater the proportion which is "passive," but if the particles are in contact with one another they all rust. Again, in a solution of ammonia and ammonium chloride pieces of soft iron may either dissolve readily or remain unattacked. "Passivity" in this solution is produced by heating until temper colours appear, or often merely by cleaning with coarse emery and exposing to the air. The metal is activated by contact with zinc or active iron in the solution or by cathodic treatment at a sufficiently negative potential. Any of these activating agencies will also cause mill-scale or ferrous oxide to dissolve in this solution, so that the observed passivity of iron is ascribed to a superficial oxide film. The progressive corrosion of iron in the presence of air and natural waters may be explained without assuming the intervention of carbon dioxide. Electrolytic action will produce at anodic areas in the presence of oxygen a coating of ferric hydroxide containing within its pores ferric chloride and hydrochloric acid (derived from sodium chloride), both of which attack the underlying metal chemically. Since sodium hydroxide produced at cathodic areas is readily washed away, excess of iron salts spreads over the surface and electrolytic action is displaced by chemical corrosion by oxygen with these salts as intermediaries. Experimental evidence is cited in support of this theory.
H. J. T. Ellingham.

Properties of electrolytic iron. G. P. Fuller (Trans. Amer. Electrochem. Soc., 1926, **50**, 189—195).—Electrolytic iron manufactured by the Niagara Electrolytic Iron Co., using the Boucher–Bouchayer process, contains, after annealing to remove hydrogen: 99·965% Fe, 0·015% Cu, and about 0·005% each of C, Si, S, and P, and has a scleroscope hardness 14—16, a yield point of about 23,000 lb./sq. in., and a tensile strength of about 40,000 lb./sq. in. The metal can be annealed at a high temperature and quenched at once in cold water without appreciably changing its properties. Tubes about 13 ft. long and 9·5 in. diameter are deposited directly and cold-worked to other diameters and wall thicknesses: for a 2·5 in. diameter tube the walls may be as thin as 0·02 in. The high resistance of electrolytic iron to chemical corrosion is further increased by cold-working.
H. J. T. Ellingham.

Corrosion of steel by acids. V. Duffek (Korrosion u. Metallschutz, 1926, **2**, 152—154; Chem. Zentr., 1926, **2**, 1328—1329).—The author worked with 2% solutions of sulphuric, nitric, hydrochloric, and oxalic acids at 20°, immersing special steels for 24 hrs. in 30 c.c. of the acid, and using test pieces $10 \times 10 \times 20$ mm. Highly polished steels are attacked less than rougher samples, but for accurate results the solution must be agitated, and the author concludes that several samples should not be placed in the liquid together because of the influence of solubility. The test pieces should be of the same shape and have the same surface area. W. G. Carey.

Effect of manganese, silicon, and phosphorus on the pearlite interval. B. Kjerrman (Trans. Amer. Soc. Steel Treat., 1926, **9**, 430—451).—The pearlite interval of a number of steels made by different processes was studied by the electric resistance method. In the absence of manganese, silicon, or other alloying element Ac1 occurred at a definite temperature or over a very small range. Anomalies were found which were thought to be connected with non-metallic inclusions, as crucible steel showed smaller intervals. Manganese depressed the beginning of the interval and widened the range. Silicon raised the interval and extended it. In a silicon–manganese steel the interval was doubled. Heat treatment tending towards homogeneity of structure reduced the range caused by manganese but did not affect the range caused by silicon. The effect of these two elements is explained on the assumption that at the same temperature manganese and silicon are respectively more and less soluble in austenite than in ferrite. The behaviour of phosphorus was similar to that of silicon.
T. H. Burnham.

Dependence of the electrical resistance of nickel steel on composition, temperature, and heat treatment. F. Ribbeck (Z. Physik, 1926, **38**, 772—787, 887—907).—After quoting figures showing the lack of agreement obtained by other workers for nickel steels, the author describes his method of determining the resistance and gives his results for iron as pure as possible, 10·10 microhms/cm., corrected for impurities to 9·62; a table gives the variation in resistance up to 1000°. For

the purest nickel obtainable the resistance was 10·6 microhms/cm., with an uncertain correction to be made for impurities; the variation up to 500° is given. Tables are given showing the resistances of steels containing various proportions of nickel and carbon for a range of temperature from about 20° to 300°. To obtain the samples as nearly as possible in a condition of equilibrium they were heated at 800° for an hour and then slowly cooled. Some steels, however, showed an "induced effect" ("Anlasserscheinung"), possessing a lower resistance when cooled from 300° to a given temperature than when heated to that temperature. One steel, with 9·55% nickel and 0·59% carbon, was so hard that it could not be turned on the lathe; it became rather softer after heating at 500° for an hour followed by slow cooling. Reversible and irreversible steels (cf. Osmond, B., 1899, 278) are discussed. In all cases the resistance increases with rise in temperature (unlike certain nickel–chromium alloys), the temperature coefficient being about 0·075 microhm/cm./degree. With increasing nickel content the resistance increases rapidly at first, then slowly from 5% to 20%, then again more rapidly; with 29·9% of nickel the resistance is eight times that of pure iron.

E. B. LUDLAM.

Dilatometric method of heat treatment. O. E. HARDER, R. L. DOWDELL and A. C. FORSYTH (Trans. Amer. Soc. Steel Treat., 1926, **9**, 403—419).—A dilatometric heat-treatment furnace is described in which a fused silica rod rests vertically on the steel to be hardened, and the expansion of the steel operates the movable plunger of a dial indicator. The specimen is heated a few divisions above the arrest point and then quenched. By this device tools and dies were more accurately hardened as regards final size than by other methods. For the investigation of cast iron a dilatometer was constructed in which two alined silica push-rods touch the ends of the specimen placed horizontally in the heating furnace. One rod works against the diaphragm of a mercury reservoir fitted with a capillary tube indicator, the other being connected to a micrometer arrangement. During heating the level of the mercury was maintained constant by adjusting the micrometer, the dilation being read off directly on the micrometer dial.

T. H. BURNHAM.

Welding of high-chromium alloys intended to meet extreme conditions. S. M. NORWOOD (Trans. Amer. Electrochem. Soc., 1926, **50**, 133—136).—Brittleness caused by welding alloys containing more than 10% of chromium is considered a defect inherent in the alloy rather than due to the method of welding. The addition of about 8% of nickel to high-chromium alloys has been found beneficial since it refines the grain and improves the ductility and shock-resistance of the alloy. To counteract the tendency to diminish the resistance of the alloy to corrosion by sulphur gases, the silicon content is increased to about 2%. It has also been found that if the manganese content is made approximately equal to that of the silicon a flux-covered rod becomes unnecessary, the products of oxidation being self-slagging.

C. A. KING.

Voltage in copper refining cells. C. G. FINK and C. A. PHILIPPI (Trans. Amer. Electrochem. Soc., 1926, **50**, 125—131).—The fall of potential in copper-refining cells, where two copper electrodes are immersed in a dilute sulphuric acid–copper sulphate bath, depends on the fall of potential through the solution and on polarisation of the anode and cathode. Variation of these factors with changes in temperature, acid and copper concentrations of the bath, has been investigated. Rising temperature or acid concentration lowers the potential fall in the solution and decreases the potential difference due to anodic and cathodic polarisation but, with regard to the latter factor, the effect becomes less marked as the acid concentration rises. Increase in copper sulphate concentration causes increased potential fall in the solution but reduces the polarisation effects to a diminishing extent. There is, thus, an optimum copper concentration for production of the minimum overall voltage. Introduction of gelatin into the electrolyte has no effect on the anode polarisation or solution potential fall, but causes a very marked increase in the cathode polarisation, which becomes constant after the addition of a small amount of the colloid.

L. M. CLARK.

Influence of shrinkage on the mechanical properties of alloys of copper and of aluminium. L. GUILLET (Compt. rend., 1926, **183**, 541—544).—The physical properties in the untreated and annealed states, and the degree of shrinkage due to forging at high temperatures of various alloys of copper and of aluminium have been determined. The shock tests are affected most by the degree of shrinkage and show distinct maxima, which are highest in the case of aluminium alloys. A great difference exists between the results obtained when the shock tests are applied in the same direction as and perpendicular to the lamination of the sample, and this difference increases beyond a certain degree of shrinkage. Thence the most favourable "degrees of shrinkage" are found to be 2·0 and 2·5 for alloys of copper and of aluminium respectively.

J. GRANT.

Properties of addition agents in electrodeposition. G. FUSEYA and K. MURATA (Trans. Amer. Electrochem. Soc., 1926, **50**, 87—114).—Determination of iron concentrations and of migration numbers shows that copper and silver form complex cations with metaphosphoric acid, organic hydroxy- and amino-acids. Sugars show little, if any, tendency to form such complexes; when copper or silver salt solutions, to which have been added these complex-forming materials, are electrolysed, some of the added material is deposited with the metal. It is suggested that this explains the diminution of grain-size in such deposits. No change of grain-size was noted with substances which do not form complexes, and which are not deposited with the metal during electrolysis. No formation of colloidal particles was observed on mixing silver nitrate and glycine or tartaric acid solutions, and the production of fine-grained deposits cannot, therefore, be ascribed to this cause.

L. M. CLARK.

Acid zinc plating baths. M. R. THOMPSON (Trans. Amer. Electrochem. Soc., 1926, **50**, 219—243).—The importance of the p_H value of the plating bath is indicated, the best value for acid zinc plating in the body of the solution being from 3·5 to 4·5, range measure-

ments being made with bromophenol-blue. At a lower p_H the cathode efficiency is decreased especially at low current densities, and at a higher p_H the deposits are coarse-grained and spongy. The p_H value of the cathode film probably approaches 6·2, *i.e.*, the precipitation point of zinc hydroxide. To maintain the desired p_H the solution must be buffered with aluminium salts from which the hydroxide is precipitated at or before p_H 4·5, so preventing the p_H of the solution from increasing, at least until nearly all the aluminium is precipitated. The deposits from such solutions are whiter and brighter. It was not found possible to increase materially the throwing power of an acid zinc bath as the cathode polarisation is low. The addition of dextrose, dextrin, glycerin, and β-naphthol causes a slight increase in polarisation and throwing power, giving smoother but darker deposits. The conductivity is increased by using zinc chloride instead of sulphate, and adding a large amount of sodium or ammonium chloride; thus, higher current densities can be used. Zinc deposited at low current densities tends to form isolated crystal groups but at higher values the deposits are continuous. A good plating bath was obtained by using a solution containing zinc chloride 136 g., sodium chloride 234 g., and aluminium chloride 20 g. per litre at a current density of 1·5—5 amp./dm.^{2} for articles of not too irregular shape or from 10 to 15 amp./dm.^{2} in agitated baths.

F. G. Crosse.

Equilibria between lead, oxygen, and sulphur in the roasting [of lead sulphide]. Schenck (Metall u. Erz, 1926, **23**, 326; Chem. Zentr., 1926, II., 1576—1577; cf. B., 1925, 287, 995).—The vapour pressure relationships of lead sulphide are discussed, and the existence of three basic lead sulphates is demonstrated. All these, as well as lead oxide and lead sulphate, can be converted into metallic lead and sulphur dioxide by heating with lead sulphate, if certain conditions are observed. Under other conditions, basic sulphates or lead oxide and sulphur dioxide are formed. In all, 13 equilibria are possible, of which 8 or 9 are accessible to observation.

C. T. Gimingham.

Relatively fine-grained deposits from "unsatisfactory" electrolytes. E. A. Vuilleumier (Trans. Amer. Electrochem. Soc., 1926, **50**, 83—86).—A dense, fine-grained deposit of lead is obtained by electrolysis of a lead acetate solution on a copper cathode which is covered with a film of glycerin, whereas a loose, rough coating is produced in the absence of the glycerin film. The effect is not so strongly marked when the acetate solution is acidified with nitric in place of acetic acid, nor when a concentrated sugar solution is substituted for the glycerin. A more dense deposit of silver from a nitrate solution is similarly produced by the use of a gold or silver cathode, previously wetted with distilled water or glycerin.

L. M. Clark.

Cadmium: its electrodeposition for rust-proofing purposes. C. M. Hoff (Trans. Amer. Electrochem. Soc., 1926, **50**, 269—278).—A comparison of the physical and chemical properties of cadmium with those of zinc indicates that the former should be the better for the rust-proofing of iron; it is less active chemically, forms a protective oxide film, is not amphoteric in character, is on the alkaline-earth side of iron in the electromotive series of metals, and, although softer than zinc, is more ductile. A satisfactory plating bath consists of a mixture of cadmium hydroxide, ammonium sulphate, and "gulac" dissolved in a solution of sodium cyanide and sodium hydroxide, using cadmium anodes. This solution has a specific resistance of 4·9 ohm./c.c. at 24°. The anode polarisation potential is 0·24 volt, the cathode 0·44 volt at a cathode current density of 2·7 amp./dm.^{2} The best results are obtained with an anode current density of 6·5—7·6 amp./dm.^{2}

F. G. Crosse.

Metals to resist corrosion or high temperatures. H. J. French (Trans. Amer. Electrochem. Soc., 1926, **50**, 47—81).—A *résumé* of the characteristics of metals and alloys used industrially to resist high temperatures or corrosion. The metals copper, aluminium, lead, tin, silver, nickel and iron, and their alloys, are considered. Tests on the steam erosion of metals made in Switzerland, in which a jet of steam was directed upon specimens of different metals at a stated velocity of 3600 to 3900 ft. per sec. for 70 hrs., showed that both low- and medium-carbon high-chromium steels, *e.g.*, stainless steel and iron, possessed favourable characteristics, but the best result was obtained with high-carbon nickel steel which also contained appreciable proportions of chromium, silicon, and manganese.

C. A. King.

Refractories for melting pure metals: iron, nickel, platinum. L. Jordan, A. A. Peterson, and L. H. Phelps (Trans. Amer. Electrochem. Soc., 1926, **50**, 115—124).—Crucibles made from commercial fused oxides, alumina, zirconia, and magnesia, were unsuitable for melting pure iron, the alumina and zirconia because of contamination of the melt by silicon and the magnesia because of sulphur. Strong and dense crucibles can be made from calcined, chemically pure magnesia mixed with 2% of magnesium chloride and about 10% of water, and such crucibles fired to about 1600° enable pure melts to be obtained. Magnesia crucibles bonded with shellac and alcohol are also satisfactory, and materials found suitable for melting pure iron were satisfactory also for nickel. Crucibles made of zirconia freed from carbon and iron soluble in acid proved suitable for melting pure platinum and platinum–rhodium alloys, providing the melting is done under oxidising conditions. Electrically sintered zirconia is extracted with hydrochloric acid (1 : 1), roasted, and prepared as a casting slip by grinding with about 4% of kaolin. Crucibles are then cast, dried, and fired carefully to 1700°. Although hard and strong, zirconia crucibles do not withstand sudden temperature changes, and are best used with an outer supporting crucible of alundum or porcelain.

C. A. King.

Failure of metals by fatigue. H. F. Moore (Trans. Amer. Soc. Steel Treat., 1926, **9**, 539—552).—When slip occurs in a metal crystal due to applied stress, the crystal splits into laminæ in a direction determined by the planes of weakness in the grain. Under repeated stresses the serrated edges of the crystal and the roughened surfaces of the laminæ behave like minute files. The result of slip under alternating stresses is the net balance of two actions, an increase in the resistance to slip, and a gradual filing action tending to start and spread

cracks. After many cycles at low stresses, mild steel resists reversed stresses many times better than if initially unstressed. Cold work by tensile stress lowers the resistance to repeated stress, but rolling packs the crystalline grains closer and tends to prevent the filing action due to slip. T. H. Burnham.

Patents.

Production of cast iron. Meier & Weichelt (E.P. 234,106, 12.5.25. Conv., 13.5.24).—Instead of producing the graphite in cast iron by means of the secondary decomposition of cementite, in the form of laminæ or veins, a finer grade of cast iron is obtained, consisting of mixed crystals and a eutectic mixture of graphite and iron, by taking an ordinary iron from a cupola furnace (Si about 3·5%), and rapidly cooling in an iron mould. The rate of cooling may be reduced in proportion as the carbon content of the iron is lower. The eutectic graphite so produced is distributed in fine particles, the size of which is no larger than the breadth of the finest striations of lamellar pearlite, and is especially useful for the production of high quality grey iron having a tensile strength of 30—40 kg./mm.^{2} and bending strength of 50—80 kg./mm.^{2}. If, however, a little white iron be also produced, this is easily broken up by short heating at about Ac1, when the cementite is very rapidly decomposed into ferrite and temper carbon. F. G. Crosse.

Production of copper from spent pyrites. F. Curtius & Co. (G.P. 431,387, 31.1.25; see G.P. 427,011). —The burnt ore is leached, and when dry subjected to magnetic separation. The magnetic portion is treated for copper by subjecting to a chloridising roast, or by heating with fresh pyrites, according to G.P. 427,011 (cf. E.P. 230,415, B., 1925, 597), while the non-magnetic fraction is used direct for the production of iron. The additional preliminary leaching is preferable to magnetic separation only. W. G. Carey.

Depositing copper from solutions. Orkla Grube Akt. (E.P. 248,724, 16.2.26. Conv., 6.3.25).—Copper from non-metallic ore products, such as pyrite cinders subjected to chlorination roasting, is recovered by leaching them and depositing the copper from solution by means of scrap brass or other alloy of copper rich in zinc. F. G. Crosse.

Imparting various colours to brass articles. F. A. E. Stahlschmidt (E.P. 258,796, 24.3.26).—The articles are immersed in a cold solution of potassium hydroxide, potassium sodium tartrate, and copper sulphate, the time of immersion determining the final colour of the articles. A final treatment with dilute acid produces a golden appearance. F. G. Crosse.

Lead and silver from lead–zinc sulphide ores. E. Langguth (G.P. 430,919, 1.1.25).—The recovery of lead and silver from sulphide ores by treatment with zinc in a melt of zinc chloride and alkali chloride is modified by the addition of alkaline-earth chlorides, or such alkaline-earth compounds as will be converted into chlorides by the zinc chloride, to the fluxing salts or to the ore, in an amount at least equivalent to the sulphate content of the ores. The high consumption of zinc due to the sulphate content of the ores before subjection to the froth flotation process is thereby reduced.

Lead alloys. C. T. J. Vautin and C. V. Stephens (E.P. 257,676, 5.6.25).—An alloy of lead containing 0·1—2·0% of cadmium is used for extruded metal work, an alloy of such composition forming no eutectic which segregates on solidification from the molten state. The alloys are stronger and less corrodible than lead itself; their uses include the production of composition piping and sheathing for electric cables. C. A. King.

Decomposition of chromium ores. I.-G. Farbenind. A.-G., Assees. of R. Caspari (G.P. 431,644, 23.3.24).—The ore, mixed with an alkaline material, is spread in a thin layer, and is exposed without agitation to the action of oxidising gases. W. G. Carey.

Preparation of alloys [of beryllium and silver]. Beryllium Corp. of America, Assees. of H. S. Cooper (E.P. 257,473, 21.12.25. Conv., 2.12.25).—The introduction of beryllium into an alloy of silver increases the resistance of the alloy to tarnishing in a marked degree. Such alloys may comprise beryllium and silver only or may contain up to 10% of each metal. Alloys of this character are made by electrolysing a beryllium salt using a cathode of molten silver. C. A. King.

Concentration of ores containing metals of the platinum group. Minerals Separation, Ltd., and J. C. Moulden (E.P. 258,648, 22.6.25).—In the froth-flotation concentration of an aqueous pulp of ore containing metalliferous matter and gangue in the presence of a protective colloid (*e.g.*, starch, glue, gelatin, etc.), better separation may be secured by adding alkaline xanthate to the ore pulp so that it is present with the colloid. A Norite sulphide ore from South Africa which before treatment assayed 0·21% Ni, 0·11% Cu, and 2·1 dwt. of platinum metals per ton, gave a concentrate containing 73·7 dwt. of platinum metals, 7·68% Ni, and 3·75% Cu. F. G. Crosse.

Preventing formation of crystals and deposition of incrustations on metal surfaces. Antiscale Co., Ltd. (E.P. 236,534, 15.6.25. Conv., 3.7.24).—The walls of the containing vessel are subjected to the action of a magnetic field or weak electric current. Thus crystals form as a fine loose powder, and boiler scale as a soft pulp or sludge. F. G. Crosse.

Tungsten wire (E.P. 258,642).—See XI.

Heat treatment by electricity (E.P. 258,049).—See XI.

XI.—ELECTROTECHNICS.

Refractories for induction furnaces. M. Unger (Trans. Amer. Electrochem. Soc., 1926, **50**, 41—46).—For induction furnaces of the horizontal ring, iron-core type, operating at 1500° to 1800°, magnesium oxide proved to be the most satisfactory lining, and when properly constructed would have a life of 400 heats with slags containing 20 to 25% of silica. Proper grading of the magnesium oxide is important, and pitch was found to be the best bonding material. To obtain a lining of maximum density a mixture known as "Furnite" (U.S.P. 1,444,527, B., 1923, 310 A) was rammed carefully to form the contour of the melting crucible. Inorganic bonds, *e.g.*, magnesium salts, ball clay, sodium silicate, proved unsatisfactory. C. A. King.

Thermal insulation of electric furnaces. M. L. Hartmann and O. B. Westmont (Trans. Amer. Electrochem. Soc., 1926, **50**, 155—176).—Thermal conductivities of a high-temperature insulating refractory (B., 1925, 761), of fused alumina and fused magnesia refractories, and of fireclay, have been determined and their variations with temperature recorded on a graph, with the corresponding data for carborundum refractories (B., 1925, 549) and silica. The chemical composition, bulk specific gravity, and porosity of these materials are stated, and available data for their mean specific heats have been collected. From these figures and values for heat losses from rough, vertical, plane surfaces (Trinks, " Industrial Furnaces "), calculations are made of the temperature gradients, heat capacities, and heat losses of furnace walls of various thicknesses and inside temperatures, these walls being either of simple refractories or of refractories with an external insulation. The use of such external insulation is important with " super-refractories " such as carborundum, since otherwise heat losses can only be kept low by having very thick walls, thereby increasing the external surface and heat capacity of the walls. A high heat capacity limits flexibility of operation, especially where intermittent working is desired. The above-mentioned insulating refractory can be used up to 1450° and is therefore suitable for backing relatively thin walls of super-refractory. Such composite walls may be designed to give greatly improved energy conservation. Present methods of insulating various types of electric furnace are briefly reviewed.

H. J. T. Ellingham.

Products formed during the working of lead accumulators. L. Mazza (Atti R. Accad. Lincei, 1926, [vi], **4**, 215—218).—By means of the Debye–Hull powder method it is shown that the substances formed during the charging and discharging of lead accumula tors include pulverulent and massive lead, lead sulphate, lead peroxide, and mixtures of these substances in various proportions. T. H. Pope.

See also pages 950, **Copper-refining cells** (Fink and Philippi); **Addition agents in electrodeposition** (Fuseya and Murata); **Acid zinc plating baths** (Thompson). 951, **Fine-grained deposits from " unsatisfactory " electrolytes** (Vuilleumier); **Electrodeposition of cadmium** (Hoff).

Patents.

Tungsten wire [for electric lamps]. Edison Swan Electric Co., Ltd., and G. A. Percival (E.P. 258,642, 20.6.25).—The introduction of beryllium silicate in small quantities, not exceeding 0·9%, into tungsten wire, for the purpose of filaments in electric lamps, is found to minimise the tendency of the wire to change its crystalline structure when heated by an electric current. Colloidal solutions of pure silica and beryllium oxide are mixed in such proportions that on fusion the resultant compound is equivalent to the meta- or ortho-silicate.

F. G. Crosse.

Galvanising bath. W. A. F. Pfanhauser, and Langbein-Pfanhauser-Werke A.-G. (E.P. 259,072, 21.12.25).—In a galvanising bath of the travelling type in which articles to be treated are suspended and subsequently removed from a travelling device at substantially the same point, the electrolyte is continually pumped through a filter into the galvanising chamber in such manner that the bath liquid is agitated and anode slime removed, thus enabling a high current density to be employed to galvanise the articles during a single passage through the bath. J. S. G. Thomas.

Heat treatment [surface hardening] of materials by electricity. W. J. Brown, and Metropolitan-Vickers Electrical Co., Ltd. (E.P. 258,049, 23.7.25).—Material to be treated, *e.g.*, metal, is placed within a solenoid through which is passed an alternating electric current of such frequency that heat is developed chiefly at the surface of the material. The material is quenched while still subjected to the alternating field.

J. S. G. Thomas.

XII.—FATS; OILS; WAXES.

Olive oil production in Tunis. M. Rousseau (Bull. Mat. Grasses, 1926, 163—193).—The number of olive trees in Tunis is about 8,500,000, and is increasing; the yield of oil is irregular, with an annual mean of 300,000 quintals, Tunis being fifth in order of productivity after Spain, Italy, Greece, and Portugal. A commission under the Department of Agriculture of Tunis examined in 1923—1925 the various factors affecting the separation of olive oil from the fruit pulp and the yield and quality of the oil. The fruit was broken up in a mill, subjected to preliminary and final pressing in a modern type of mechanically-operated oil press, and the oil decanted from the pulp by some form of separator. The following results are reported :—(1) Elimination of leaves and stalks and washing of the olives are essential to the production of oil of superior quality; the type of plant to carry out these operations is briefly indicated. (2) Influence of duration of storage of olives in ordinary silos is seen in rapid deterioration of the quality of the oil by reason of fermentation set up; olives kept two days gave oil of free fatty acids 0·5%, and kept 18 days gave oil of free fatty acids 3%. (3) Under comparable conditions the results when storing in closed silos were worse than when using ordinary ones. (4) When stored on wooden screens placed one above the other stagewise, the olives yielded better oil than when stored in silos because of freer circulation of air, less internal heating, and therefore less chance of fermentation. (5) The influence of salting on olives kept for 36 days is to hinder the development of moulds on the fruit and high acidity of the oil and equally to hinder elevation of temperature of olives stored in heaps and to diminish the intensity of any fermentation. It is advisable, however, to apply salting only to those olives of poor quality or to those that have commenced to undergo change, since salting imparts an objectionable flavour to the oil. (6) When investigating the duration and fineness of the breaking or crushing of the fruit it was found that the total yield of olive oil increases with duration of the crushing; the yield of oil of first pressings increases with duration of crushing, while the yield of oil at second pressing diminishes. The quality of oil diminishes with increase in the duration of the crushing and this diminution begins to be felt when the duration is beyond 30 to 40 minutes. (7) A comparison of the methods of separating the oil from the accompanying pulp after-

pressing shows that the most advantageous is the use of the Hignette centrifuge, the Sharples centrifuge closely following in merit, whilst the automatic washing separator appears least satisfactory. Moreover, the quality of oil when using the last-named method of separation is certainly inferior to that obtained by the other methods, the acidity of first pressings oil being 0·75%, and of second pressings 1·9%, the acidity of the oil in the olives being originally 0·5%. H. M. LANGTON.

Vegetable oils containing glycerides of erucic acid. J. J. SUDBOROUGH, H. E. WATSON, and P. R. AYYAR (J. Indian Inst. Sci., 1926, 9A, 25—70).—Full analytical constants of the oils are given and the solid and liquid acids, separated by Twitchell's lead salt–95% alcohol method (B., 1921, 817 A) have been analysed by analysis of the fractionated methyl esters. The refractive index–iodine curves for the hardened oils have also been studied (cf. B., 1924, 755).

[With N. R. DAMLE.] Expression of the seeds of *Brassica campestris* var. *napus* yielded 38% of rape oil having an acetyl value of 31, due to the presence of mono- or di-glycerides. The composition of the acids from the oil was: myristic 1·5%, stearic 1·6%, behenic 0·5%, lignoceric 2·4%, oleic 20·2%, erucic 57·2%, linoleic 14·5%, linolenic 2·1%, and from the hardened oil: myristic 1·5%, stearic 38·5%, behenic 57·6%, lignoceric 2·4%.

[With V. M. MASCARENHAS.] A yield of 35·2% of Indian mustard oil ("rai") was obtained by extraction of seeds of *Brassica juncea*, D.C. The acids from the oil had the composition: myristic 0·5%, stearic nil, behenic 3·8%, lignoceric 1·1%, oleic 32·3%, erucic 41·5%, linoleic 18·1%, linolenic 2·7%, and those from the hardened oil: myristic 0·5%, stearic 52·1%, behenic 46·3%, lignoceric 1·1%.

[With T. J. MIRCHANDANI.] Appreciable differences existed between the extracted and expressed Jamba oils from the seeds of *Eruca sativa*, Lam, the latter closely resembling Indian rape oil and having an acetyl value of 22. The acids from the expressed oil consisted of: stearic 4·2%, behenic 4·5%, lignoceric 1·8%, oleic 28·7%, erucic 46·3%, linoleic 12·4%, linolenic 2·1% and those from the hardened oil: stearic 47·4%, behenic 50·8%, lignoceric 1·8%. A sulphur compound, which inhibited hydrogenation, could only be removed by hot extraction of the oil with 88% alcohol. Maceration of the seeds with water containing sodium fluoride followed by steam distillation gave an average yield of 0·8% of a volatile oil having a penetrating odour and containing sulphur and nitrogen, but differing considerably in properties from the essential oil of rape seed. Attempts to isolate the glucoside were unsuccessful.

[With N. R. DAMLE]. The seeds of *Tropæolum majus*, Linn., on extraction yielded 7·2% of oil having $d^{15.5}$ 0·9092, n_D^{40} 1·4568, iodine value 77·5, saponif. value 172·6, and unsaponifiable matter 1·1%. The mixed fatty acids had Hehner value 95·1, iodine value 72·9, and mean mol. wt. 312·8. The solid acids (45·7%) had iodine value 72·9, mean mol. wt. 330·0, and the liquid acids (54·3%) had iodine value 72·6 and mean mol. wt. 298·0. Tribrassidin (m.p. 56—57°, n_D^{60} 1·4547) and tribehenin (m.p. 81—81·5°, n_D^{85} 1·4391, n_D^{89} 1·4375) were prepared from trierucin (m.p. 30·5—31·0°, n_D^{40} 1·4630, n_D^{60} 1·4560, n_D^{81} 1·4475) crystallised from the oil. The m.p. of purified behenic acid prepared by six different methods ranged from 79·3° to 80°. The solidifying point curves of mixtures of methyl stearate and methyl behenate and the solidifying and melting point curves of mixtures of stearic and behenic acids have been determined. E. H. SHARPLES.

Oil of *Pongamia glabra*. G. D. BEAL and M. C. T. KATTI (J. Amer. Pharm. Assoc., 1925, 14, 1086—1096; Chem. Abstr., 1926, 20, 2895).—The seeds contain 27—33% of oil of a bitter taste and unpleasant odour, saponification value 189·1, iodine value (Hanus) 89·1, Reichert–Meissl value 1·04, unsaponifiable matter 2·4%, acid value as oleic acid 8·36%, soluble acids as butyric acid 1·96%, insoluble acids together with unsaponifiable matter 93·25%, neutralisation value of insoluble acids 196·1, mean mol. wt. of insoluble acids 286·0, iodine value (Hanus) of insoluble acids 92·6. Distillation in steam removes most of the odorous constituents, and the bitter principle (unsaponifiable) is extracted with 76% ethyl alcohol. A substance, m.p. 157·5°, becoming red in sunlight, constituted or contained the bitter principle. A. A. ELDRIDGE.

Effect of p_H on the germicidal action of soaps. A. H. EGGERTH (J. Gen. Physiol., 1926, 10, 147—160).—The action of various fatty acids (butyric, caproic, capric, caprylic, undecoic, lauric, tridecoic, myristic, pentadecoic, palmitic, stearic, oleic, and ricinoleic, and their salts at different hydrogen-ion concentrations on a number of bacteria (*Streptococcus pyogenes*, *B. diphtheriæ*, *Staphylococcus aureus*, *B. typhosus*, and *Vibrio choleræ*) depends both on the acid and the particular bacterium used, but in general the lower acids of the series are more active at an acid reaction, whereas the higher numbers are more active at an alkaline reaction. W. O. KERMACK.

PATENT.

Manufacture of soaps. A. H. BAILY and J. N. KIRBY (E.P. 256,362, 6.6.25).—Hard soaps particularly suitable for degreasing fibres and for the degumming of certain vegetable fibres are made by adding an alkali protein solution to the usual mixture of saponified fatty oils. The protein solution, made by dissolving caseinogen or the like in excess of alkali and adding 0·5—1·0% of formalin, is added in quantities as required to give the desired quality of soap. Similarly, the protein solution may be used with the desired combination to give liquid soaps and dry soaps. H. M. LANGTON.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Importance of particle properties in paint pigments. C. A. KLEIN (J. Oil and Colour Chem. Assoc., 1926, 9, 192—196).—Both chemical and physical investigations are necessary in connexion with the problems offered by pigments. The variation and irregularity in the particle shape and size are matters of much importance to the paint manufacturer. The presence of oversized particles has an effect disproportionate to their number. Much has still to be learnt as to the effects produced by so-called "inert extenders." Various problems awaiting attention are indicated.

D. F. TWISS.

Influence and elimination of coarse particles [from pigments]. N. HEATON (J. Oil and Colour Chem. Assoc., 1926, 9, 215—218).—The particles of pigments can be conveniently classified into three groups, viz., "coarse"—over 60 microns in diameter, "intermediate"—between 10 and 60 microns, and under 10 microns. In most pigments the intermediate and coarse particles are present only in small percentage. Probably the most difficult pigments to obtain in a satisfactory degree of fineness are those prepared by successive precipitation and calcination. D. F. TWISS.

Use of asbestine (micro-asbestos) in the paint industry. H. ROSENBERG (Farben-Ztg., 1926, **31**, 2936).—The properties of European and American asbestine are discussed, the latter being almost pure magnesium silicate, whilst the composition of a typical sample of the former is as follows:—silica 49·81%, alumina 2·19%, iron oxide 2·80%, magnesia 27·96%, lime 13·21%, water 3·91%, and traces of manganous oxide. Asbestine finds application in the paint industry as an inert extender, a base for lake pigments, and a desirable constituent of acid-, alkali-, and fire-resistant paints. S. S. WOOLF.

Wetting power of solvents, and their behaviour on evaporation. H. VOLLMANN (Farben-Ztg., 1926, **31**, 2932—2934).—Various solvents used in the paint and varnish industry were dropped from a capillary pipette on to brass and glass plates, and on to duplicate plates that had been washed with the particular solvent and wiped dry. The volume of a drop, extent to which it spread, its appearance and subsequent behaviour in the course of volatilisation are tabulated and discussed. Greater spreading and more rapid evaporation were found on brass than on glass. Preliminary washing of the plates increased the spreading power, by displacement of an adsorbed gas-layer by a layer of the solvent. The wetting power of solvents is dependent to some extent on their chemical structure, paraffins, for instance, showing marked differences from benzene and its homologues. S. S. WOOLF.

Rapid evaluation of baked japan finishes. E. M. HONAN and R. E. WATERMAN (Ind. Eng. Chem., 1926, **18**, 1066—1068).—It is proposed to evaluate the service life of a japan film baked on metal by immersing test pieces in an 8·5% solution of phenol in water (which has constant composition at normal room temperature) until at least half the japan has wrinkled or has peeled off. For the same japan, the time of resistance to phenol is found to depend on stoving time and temperature, nature of metal coated, and condition of the latter before coating. The method gives results consistent with those of parallel service tests. S. S. WOOLF.

Condensation of formaldehyde with phenols and urea. H. BARTHÉLEMY (Chim. et Ind., 1926, **16**, 367—372).—A discussion of the mechanism of condensation. By addition of aqueous alcohol to an alcoholic solution of the partially condensed product a mixture of two colloidal suspensions is obtained, which can be separated into positive and negative components by the action of an electric field. These suspensions, which possess the Brownian movement, are regarded as ionic miscelles having the constitutions: $[(\Sigma_1^n C_6H_4O,CH_2)H]OK$, $+ve$, $[(\Sigma_1^n C_6H_4O,CH_2)H]Cl$,—$ve$. Further condensation is influenced by the nature of the catalytic ion, solvent, and temperature. The properties and characteristics of "Prystal," a urea–formaldehyde condensation product, are described. J. S. H. DAVIES.

Painting treated wood. DUNLAP.—See IX.

Detection of grit in rubber pigments. MURPHY.—See XIV.

PATENTS.

Production of [green] pigment colours. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 257,528, 21.4.26).—The shade of the green pigment colours produced by the process described in E.P. 181,584 (B., 1922, 600 A) is influenced by the addition of basic dyestuffs, preferably of the malachite-green series, the fastness to light and alkalis being unimpaired and sometimes improved. Improvements involving the addition of Turkey red oil, or some other dispersing agent, and an alternative order of precipitation are also claimed. S. S. WOOLF.

Production of permanent pigments. DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H. (E.P. 242,282, 28.10.25. Conv., 31.10.24).—Small quantities of acid adhering to pigments after precipitation (particularly titanium dioxide) are neutralised by zinc oxide or zinc hydroxide in aqueous suspension before the filtering and drying processes. Kneading the moist pigment with zinc oxide is an alternative method of neutralisation. S. S. WOOLF.

Resinous condensation products from phenols and formaldehyde. SOC. VERRERIES FOLEMBRAY (F.P. 563,777, 27.6.22).—Mixtures of phenols with about an equal volume of 40% formaldehyde solution containing 5—10% of calcium chloride are heated until the plastic products are no longer sticky when cooled. The products when first formed are soluble in alcohol and acetone, and can readily be moulded into shape or rolled into thin sheets, or they can be mixed with rubber to accelerate vulcanisation, but on further heating or on prolonged storage at the ordinary temperature they turn hard and insoluble; they are especially adapted for the manufacture of varnish for wooden floors and ships' hulls. L. A. COLES.

Resinous condensation products from phenols and formaldehyde. J. BRUHAT (F.P. 575,532, 21.3.23).—Liquid phenol–formaldehyde condensation products are mixed with esters of polyhydric alcohols and organic acids. For example, the product obtained by condensing phenol with hexamethylenetetramine, trioxymethylene, or paraformaldehyde in the absence of a catalyst, is mixed with 10% of glycerin mono-oxalate. The product can be partially hardened by storage at 15° for several hours before it is moulded into shape, and the hardening process completed by heating, *e.g.*, at 80—100°, or it can be moulded immediately and hardened by heating to 60—100° in the air, or solutions of it in alcohol or acetone can be used as varnish. L. A. COLES.

Resinous condensation products from phenols and formaldehyde. E. ROPP (F.P. 564,575, 12.7.22).—Phosphorus pentachloride, with or without the addition

of sodium acetate, phosphorus oxychloride, or acetyl chloride, is used as catalyst in the condensations. For example, a mixture of phenol, b.p. 182—184°, purified by distillation over aluminium filings, with 40% formaldehyde or trioxymethylene free from iron, and a little phosphorus pentachloride or oxychloride, is heated to 45—50°, whereupon the temperature rises to 97°. The mixture is then heated at the boil for 20 min. After withdrawing the aqueous layer, the product is washed, heated to 100°, concentrated *in vacuo* at 160—170° until it is of the consistency of honey, and then heated to 180—190° in an autoclave for 2 hours. The product is hard and transparent, and possesses high dielectric properties. Products obtained in a similar manner from the homologues of phenol are usually cloudy. L. A. COLES.

Preparation of resinous bodies derived from acetaldehyde. L. H. BAEKELAND and A. H. GOTTHELF, Assrs. to BAKELITE CORP. (U.S.P. 1,598,546, 31.8.26. Appl., 19.12.19).—An infusible condensation product results from the reaction of a substance containing an active methylene group with a condensation product of a phenol and acetaldehyde. S. S. WOOLF.

Roll or cylinder mills for grinding paint etc. (E.P. 257,727).—See I.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Röntgen effect of stretched gels, especially of caoutchouc. W. OSTWALD (Kolloid-Z., 1926, **40**, 58—73).—Natural caoutchouc has a typically cellular structure; the cells consist of latex drops, of which the outer membranes are composed of highly polymerised hydrocarbons, whilst the gelatinised cell-contents consist of a network of uniform rod-like isoprene chains or polymerides, embedded in a dispersion medium of hydrocarbons of lower molecular weight. In the unstretched condition the distances between the meshes of the net are greater than the *X*-ray wave-length, but with sufficiently great stretching in one direction, a spindle-shaped or cylindrical latex cell may be formed in which (*a*) the net meshes occupy a uniform three-dimensional arrangement in space; (*b*) the rod-like micelles forming the net approach one another until they come within the range of the *X*-ray wave-length; and (*c*) the net, previously not necessarily rigid, becomes an immovable lattice by the strong tension on the cell membrane. The latex cell thus becomes a crystal lattice body. The theory is confirmed by the quantitative observations of Hauser and Mark (cf. B., 1926, 334, 761).
L. L. BIRCUMSHAW.

[X-Ray] interference phenomena in stretched rubber. S. REINER (Gummi-Ztg., 1926, **41**, 133—135).—A review of recent investigations and theories as to the structure of rubber, with especial reference to the results of *X*-ray spectrographic research. D. F. TWISS.

Has caoutchouc been synthesised? J. R. KATZ (Koll.-Chem. Beih., 1926, **23**, 344—348).—Synthetic caoutchouc prepared by the polymerisation of isoprene has not the same elasticity as the natural substance. On freezing in liquid air and then smashing by a blow, the cleavage is quite different. *X*-Ray examination of strongly stretched natural caoutchouc reveals a crystalline structure caused by parallel arrangement of the particles. No such structure is revealed by *X*-ray examination of synthetic caoutchouc. E. S. HEDGES.

Influence of particle size in rubber manufacture. S. S. PICKLES (J. Oil and Colour Chem. Assoc., 1926, **9**, 204—207).—The influence of particle size of compounding ingredients on rubber is discussed, with special reference to varieties of litharge, zinc oxide, and carbon produced by different processes. D. F. TWISS.

Particle shape [of powders for incorporation in rubber]. P. SCHIDROWITZ (J. Oil and Colour Chem. Assoc., 1926, **9**, 208—210).—A review of the effect of the shape of the particles of a rubber ingredient on the development of "calender grain" in the manufactured article. D. F. TWISS.

Particle size effects in rubbers subjected to repeated stress. T. R. DAWSON (J. Oil and Colour Chem. Assoc., 1926, **9**, 211—214).—Mixtures of a parent stock (100 vols.), containing rubber (95 pts. by weight) and sulphur (5 pts.), with various fillers (20 vols.) were submitted to comparative tests. The effect of the powders (barytes, clay, ordinary and "colloidal" zinc oxide, magnesium carbonate, lamp black, and gas black) on the tensile strength and resistance to extension is greater the smaller the particle size; the hysteresis (at 260 stresses per min.) is more marked the smaller the particles, as also is the scleroscope hardness. Volume increase under strain, however, is only approximately related to particle size, the degree of adhesion of the particle to the rubber being also probably a serious factor. D. F. TWISS.

Importance of particle character in a rubber "pigment." D. F. TWISS (J. Oil and Colour Chem. Assoc., 1926, **9**, 197—203).—A summary of the various effects produced by different finely-divided compounding ingredients in rubber, and of the particle features associated with these effects. The abrasion resistance of vulcanised rubber containing carbon black is affected in a less degree than the tensile strength by the presence of coarse particles in the carbon black; the "resilient energy" of such a mixture, therefore, does not supply a simple measure of the abrasion resistance.
D. F. TWISS.

Detection of grit in rubber pigments. E. A. MURPHY (J. Oil and Colour Chem. Assoc., 1926, **9**, 219—225).—A modified elutriator permitting the economical use of special media for the avoidance of incomplete dispersion, is described. For most powders a solution containing 0·5% of sodium hydroxide and 0·1% of glue in distilled water is advantageous. The usefulness of an elutriator, however, is limited, and for most purposes the sieving apparatus of Gallie and Porritt (p. 935) offers advantages. D. F. TWISS.

Origin of [X-ray] interferences in the stretching of rubber. E. A. HAUSER (Z. Elektrochem., 1926, **32**, 463—467).—See B., 1926, 638.

PATENTS.

Concentrating caoutchouc latex etc. K. D. P., Ltd. (E.P., 243,016, 13.11.25. Conv., 14.11.24).—Natural, preserved, or vulcanised latex of rubber, gutta-percha, balata, or analogous materials is dehydrated in

a horizontal rotating cylindrical chamber by the action of a current of heated air or other gas, which is first led to the bottom of the chamber so as to effect initial contact with the bulk of the latex ; evaporation is aided by rotary discs, concentric with the chamber, which dip in the bulk of the latex and continually expose fresh surface films to the action of the gas. With this method of procedure frothing and skin formation are avoided. D. F. TWISS.

Utilisation of rubber-bearing plants. F. T. LAHEY (U.S.P. 1,597,807, 31.8.26. Appl., 25.4.23).—The material of a rubber-bearing plant is reduced to a plastic mass, dried, and vulcanised. D. F. TWISS.

Process for devulcanising vulcanised rubber. C. F. WILLARD (U.S.P. 1,598,470, 31.8.26. Appl., 4.1.26).—Vulcanised rubber, together with fibrous material, is heated with a solution of an emulsoid colloid, the fibre being concurrently converted into colloidal cellulose; the mixture of devulcanised rubber and cellulose constitutes a product of superior quality and increased yield. D. F. TWISS.

Vulcanisable plastic material. F. T. LAHEY (U.S.P. 1,597,808, 31.8.26. Appl., 18.10.23).—A vulcanisable plastic material is produced by milling ground vulcanised rubber, and concurrently introducing rubber latex and emulsified oils. D. F. TWISS.

Manufacture of rubber with innumerable microscopically small pores. H. BECKMANN (E.P. 257,561, 3.8.26. Conv., 1.9.25 ; cf. E.P. 240,430, B., 1925, 1000). Latex mixed with sulphur or other vulcanising agent is coagulated by means of a manganese salt, particularly manganese sulphate; the coagulum forms a firm jelly which may be vulcanised in the manner described previously. The coagulant is removed by washing either before or after vulcanisation. D. F. TWISS.

Caoutchouc compositions. A. BIDDLE (E.P. 257,718, 31.7.25).—Latex of rubber, gutta-percha, balata, or similar substances, mixed with tapioca meal, with the addition of casein and filling materials if desired, is of use in the manufacture of insulating materials, adhesives, binding materials, etc. D. F. TWISS.

XV.—LEATHER ; GLUE.

Rôle of colloid mills in utilising tanning materials in the tanning of skins. U. J. THUAU (J. Soc. Leather Trades Chem., 1926, **10**, 258—263).—The difference between the results obtained by tanning hides with liquors extracted from the tanning material, *e.g.*, oak bark, quebracho wood, and liquors prepared from extracts of these materials is due to the agglomeration of colloidal tan (cf. B., 1923, 367). The concentration of the weak tan liquors results in the aggregation of the coarser colloidal particles and consequent precipitation when diluted again. When divi divi powder containing 21·9% of insoluble matter was powdered in a Kek ("colloid") mill (7500 r.p.m.), a 20% colloidal solution of the product contained 1·8% of insoluble matter and 7·1% of tans; after mixing the treated powder with water and passing it through the mill, the insoluble matter was 2·3%, and the tan 8·0%; after keeping the mixture for 8 days its tannin content had fallen to 6·4%. When the divi divi powder was bruised in a mortar the solution obtained contained only 1·3% of insoluble matter, but the clear liquor obtained after keeping for 8 days contained only 4·0% of tans. Similar tests on mimosa bark and sumac are described. D. WOODROFFE.

Determination of the degree of tannage [of leather] by means of the water-resistance test. Influence of drying on the water-resistance of hide powder. O. GERNGROSS and R. GORGES (Collegium, 1926, 391—397).—The water-resistance of a leather is defined as the percentage of hide substance in a leather which remains undissolved after treatment with boiling water for 7 hrs. The equivalent of 1 g. of dry hide substance is weighed into a flask fitted with a stirrer, and 100 c.c. of boiling water are poured into the flask, which is kept at 100° in a water bath for 7 hrs. The liquor is then strained through linen, 2 c.c. of the filtrate are analysed by the micro-Kjeldahl process, and the percentage of dissolved hide substance is determined. The percentage of undissolved hide substance, *i.e.*, the water-resistance, is found by difference. Drying hide powder at ordinary temperatures increased the water-resistance slightly, but drying at 110° for 24 hrs. increased it from 2 to 41. D. WOODROFFE.

Effect of hydrogen-ion concentration and neutral salts on the intensity of formaldehyde tannage. O. GERNGROSS and R. GORGES (Collegium, 1926, 398—408).—Hide powders of p_H 3·0 to 12·0 were tanned for 5 hrs. with 0·95% formaldehyde solutions, and the intensity of the tannage was determined by the water-resistance figure (cf. preceding abstract). There was scarcely any tanning effect at p_H 3·0, but it increased gradually to p_H 6·3. The isoelectric point had no significance for the formaldehyde tannage. Between p_H 6·3 and 7·0 there was a considerable sudden increase in the tanning intensity. Above p_H 7·0 the tanning intensity increased slightly to p_H 8·0, from which point it was practically constant. A four-fifths saturated sodium chloride solution and N- and 0·1N-solutions of potassium thiocyanate diminished the degree of tannage in alkaline solutions, whilst the sodium chloride had no effect in acid solutions. Experiments with hides and sheepskins confirmed the bad effect of formaldehyde tanning in strongly alkaline solutions. The degree of tannage was high, but the fibres were overswollen, and there was a kind of dead tannage. The tanning of sheepskins with formaldehyde in strong salt solutions failed in acid solutions, but resulted in a very good leather in strongly alkaline solutions (p_H 11·2 before, 8·6 after the tannage), and the leather had a water-resistance of 87. An after-treatment of this leather with egg yolk did not alter this figure. D. WOODROFFE.

Liming with sulphide. V. CASABURI (Boll. Uff. Ind. Pelli, 1926, **4**, 236—259).—The theory of the treatment of hides with alkaline sulphide is discussed and the rationale of this treatment when applied in the case of sole leather, kid, glove leather, etc., considered in detail. T. H. POPE.

Measurement of hydrogen-ion concentration in its applications to the tanning industry. G. BALDRACCO (Boll. Uff. Ind. Pelli, 1926, **4**, 227—235).—A

summary of the uses to which a knowledge of the hydrogen-ion concentration is applied in tanning process. The colorimetric method of measurement is recommended.

T. H. POPE.

Adsorption of acids by hide in connexion with swelling phenomena. I. P. N. PAVLOV (Kolloid-Z., 1926, **40**, 73—90).—From the change of solution concentration occurring on the adsorption of mineral acids by hide, the apparent adsorption isotherm can be determined, the concentration change being caused by the taking up of both solvent and solute by the hide. Simultaneous observations of the change of solution concentration and of the adsorbent swelling lead to the determination of the true adsorption of the solute. The adsorption coefficient of hydrochloric acid, sulphuric acid, and nitric acid is independent of the bath volume and of the amount of hide; from this it is inferred that the adsorption process consists in the formation of solvates of the acid molecules and ions with the collagen. The apparent adsorption isotherms of the acids form a curve with two branches. The swelling curve of hide in hydrochloric acid shows two minima, corresponding to two isoelectric points. The isotherms of the apparent "dry" adsorption and the swelling curve are comparable, a rise in the adsorption curve corresponding to a rise in the swelling curve, whilst the horizontal, or almost horizontal, branch of the adsorption curve corresponds to a fall in the swelling curve. Calculation of the true adsorption of the acids shows that, in the case of hydrochloric and sulphuric acids, the process follows the usual adsorption isotherm, and may be expressed by the Boedecker equation. With nitric acid, the true adsorption isotherm has not the simple form of those of the other acids, perhaps owing to the higher dissolving power of the acid for the collagen. The acids are adsorbed by hide in the order $H_2SO_4/2 > HCl > HNO_3$.

L. L. BIRCUMSHAW.

Extraction of chromium from leather. L. MASNER and N. J. BERESTOVOJ (Chem. Listy, 1926, **20**, 468—469).—The degree of extraction of chromium from leather by means of Rochelle salt depends on the reaction of the medium. Using sufficient of a 2·5% solution of Rochelle salt to give 2 g. of the salt for every gram of leather, a minimal extraction is found at p_H 3·58. In order to avoid hydrolysis of the leather it is recommended that solutions of moderate acidity be used. When extraction is carried out by sodium hydroxide, the hydrolysis is considerable and the amount of chromium extracted is approximately proportional to the amount of leather entering into solution.

C. RANKEN.

Treatment and disposal of waste [tannery] waters. J. NOYER (J. Soc. Leather Trades Chem., 1926, **10**, 263—265).—The waste liquors from an alum tawing works are alkaline. They contain protein matter, lime, and traces of sulphide. The settling tank has an inclined floor, and is divided into two parts, and the hair, flesh, and a little lime settle out here. The decanted liquor is agitated in a purification tank, divided into three compartments, with 50 g. of ferrous sulphate, 100 g. of sulphuric acid, 10 g. of aluminium sulphate, and 5 g. of sodium hypochlorite per cubic metre, and agitated. The ferrous sulphate forms ferrous sulphide and ferrous hydroxide, which is oxidised to ferric hydroxide and coagulates the protein. The treated liquor is filtered through coke, and if necessary softened with permutit. The liquor is then neutral, clear, and odourless. The sludge contains 5—6% of nitrogen, and has manurial value.

D. WOODROFFE.

Water content of glue and its importance in glue valuation. F. BAUM (Chem.-Ztg., 1926, **50**, 649—651, 691—692, 742—745).—The inadequacy of chemical analysis alone in judging the quality of a glue is discussed, and the behaviour of different grades on treatment with water described. Discrepancies in analysis are due partly to the difficulty of obtaining a representative sample, which is obviated by the preparation of a uniform glue jelly from which portions are taken for the various determinations, and also to the extreme slowness with which a solid glue attains the thoroughly air-dried condition. It is important to make determinations both of the total water and of that lost on air-drying. The water content of the glue in the completely air-dried condition is related to the quality, and in good varieties is about 11%. The composition of a glue may be expressed thus:—the essential air-dried glue substance with its combined water + the soluble matter lost during soaking + the excess of water removed by air-drying. The last-named constituent may vary considerably, and the marketing of glue on the basis of a standard air-dried condition is recommended.

F. R. ENNOS.

Analysis of lactic acid. VIDAL.—See XX.

PATENT.

Tanning. J. R. GEIGY SOC. ANON., Assees. of CHEM. FABR. HALTINGEN, JUCKER & Co. (E.P. 256,628, 2.9.26. Conv., 5.8.25).—Hides or skins are treated with hydrofluosilicic acid or a salt thereof prior to or simultaneously with the tanning operation. This treatment is said to facilitate the tanning process.

D. WOODROFFE.

XVI.—AGRICULTURE.

Origin and nature of soil organic matter or soil "humus." II. Method of determining humus in the soil. S. A. WAKSMAN (Soil Sci., 1926, **22**, 221—232; cf. B., 1926, 892).—Preliminary treatment with hydrochloric acid does not increase the amount of "humus" extracted by subsequent treatment with alkaline solutions. Sodium hydroxide solution is preferable to ammonium hydroxide for humus extraction. The method recommended for the determination of humus consists in heating the soil with 2·5% sodium hydroxide for 30 min. at 15 lb. pressure in an autoclave, or allowing it to react for 48 hrs. in the cold, and filtering. Fresh sodium hydroxide is added and filtration and washing are continued till the filtrate is no longer coloured. The combined filtrates are precipitated with excess of warm 10% hydrochloric acid. The precipitate (α-fraction) after collection and warming is dried at 65—70° for 12—24 hrs. and weighed. Ash and nitrogen are determined in portions of the precipitate. The filtrate from the α-fraction is treated with 2—3% sodium hydroxide solution, until maximum precipitation occurs, to obtain the β-fraction.

G. W. ROBINSON.

Absorption of iron by soils. H. C. DOYNE and C. G. T. MORISON (Soil, Sci., 1926, **22**, 163—173).—Data

illustrating the absorption of iron from ferric chloride solution are given for a number of different types of soil. Four factors appear to be concerned in the absorption of iron, namely, calcium carbonate, gross amount and activity of the clay present, ferric oxide, and organic matter. Calcium carbonate acts by neutralising the free hydrochloric acid resulting from the hydrolysis of ferric chloride. The iron is then precipitated as ferric hydroxide. Free hydrochloric acid is also neutralised by clay and there is a mutual flocculation of ferric hydroxide and clay. Ferric hydroxide gel acts simply by neutralisation. A number of curves are given to show the effect on the absorption of iron by different soils of adding varying amounts of calcium carbonate. In the case of sandy soils there appears to be a maximum absorption of iron which cannot be exceeded until sufficient calcium carbonate has been added for complete precipitation. The form of the curves obtained is discussed.

G. W. Robinson.

Loss of nitrates from cropped soils. J. Hendrick (Agric. Progress, 1925, **2**, 69—71; Chem. Abstr., 1926, **20**, 3056).—Crops appear to take up nitrates practically as fast as they are formed from the nitrogenous fertilisers applied, since no appreciable loss was observed, even in very wet weather. Drainage waters from uncropped soils contained 3—7 times as much nitrogen as those from cropped soils.

A. A. Eldridge.

Micro-organisms concerned in the decomposition of cellulose in the soil. S. A. Waksman and C. E. Skinner (J. Bact., 1926, **12**, 57—84; Chem. Abstr., 1926, **20**, 3056).—The amount of cellulose decomposed in soil under aerobic conditions depends on the amount of available nitrogen; under anaerobic conditions much less nitrogen is required. In normal soils, organisms causing anaerobic cellulose decomposition are scarce. There is a direct correlation between cellulose decomposition, the development of fungi, and the transformation of soil nitrogen into microbial protoplasm. Nitrogen-fixing bacteria probably do not increase the store of soil-nitrogen under aerobic conditions, especially in humid soils, when cellulose and straw are added as sources of energy. Nitrogen fixation occurs when starches and lower carbohydrates are also introduced.

A. A. Eldridge.

Agricultural chemical investigations. F. Münter (Landw. Jahrb., 1926, **64**, 65—127; Chem. Zentr., 1926, **97**, II, 1569).—(1) In vegetation experiments, the application of large amounts of nitrogen increased the yield of dry matter and of protein in leguminous plants. This is contrary to the results of field experiments. A sufficient supply of water appeared to be decisive. In a dry period, salts containing nitrogen act unfavourably on young lupins by making the salt concentration in the soil too high. (2) It is advantageous to plough in fertilisers on heavy deep soils. (3) In the presence of ammonium sulphate, the uptake of phosphoric acid by plants is increased by adding potassium salts, but not by adding sodium nitrate. (4) "Cave" phosphate, discovered by Willner, has little manurial value. (5) Addition of lime about six months after manuring with superphosphate increased the yield of sugar beet by 50%; lime given eight months and more after superphosphate had no effect. (6) Of the newer phosphate fertilisers, Rhenania phosphate showed the highest percentage of P_2O_5 utilised by plants. (7) Sugar beet and barley tolerate a relatively alkaline soil reaction; carrots, potatoes, red clover, flax, and yellow lupins are more sensitive. Sulphur, calcium sulphate, and manganous sulphate decrease alkalinity and increase yield. The existence of two optima for barley was confirmed—p_H 4·8 and p_H 7·6. Calcium cyanamide acts best, and sodium nitrate badly, on strongly acid soils. Acid soils are specially unfavourable in dry periods. (8) Seed stimulants are not likely to be useful in Germany since the arable soils are generally sufficiently supplied with salts. (9) Japanese results with Asahi Promoloid are not confirmed. (10) Barley, flax, and clover proved to be the best crops in regard to their influence on a following crop; sugar beet was the worst.

C. T. Gimingham.

Manuring of potatoes. Lamberg (Z. Pflanz. Düng., 1926, **B5**, 380—382).—From considerations based on Mitscherlich's theory, it is concluded that applications of large amounts of nitrogen would be justified by the increased yields of potatoes, whereas this would not be the case with large applications of potassium salts. The collated results of a large number of experiments confirm this conclusion.

C. T. Gimingham.

Effect of nitrogenous manuring of grass land on the proportion of grasses and clovers. W. Jessen (Z. Pflanz. Düng., 1926, **B5**, 394—396).—With reference to a paper with this title by Maiwald (*ibid.*, **B4**, 531), it is pointed out that the subject was previously investigated by Lemmermann, and his conclusions are quoted (cf. Landw. Vers.-Stat., 1907, **67**, 207).

C. T. Gimingham.

Manuring straw crops with increasing amounts of sodium nitrate. Kuhnert (Z. Pflanz. Düng., 1926, **B5**, 386—389).—In plot experiments with a mixture of summer rye and oats, the highest application of sodium nitrate tried gave a paying increase of yield. The experiments have reference to the difficulty of obtaining satisfactory crops of oats in certain parts of Schleswig-Holstein.

C. T. Gimingham.

Field experiment on the effect of an acid soil reaction on different [systems of] manuring. E. Möller-Arnold (Z. Pflanz. Düng., 1926, **B5**, 376—379).—A field experiment is reported on the effect of various types of manuring (acid, neutral, or alkaline) on the yield of potatoes on a very acid soil (p_H 4·0). The greatest increase of yield (83%) over that of the unmanured plot was given by an alkaline mixture of manures (sodium nitrate, basic slag, and potassium sulphate) with chalk in addition, though the acidity of the soil was not greatly reduced. The same mixture without chalk gave only an insignificant increase. An acid mixture (superphosphate, ammonium sulphate, and 40% potash salts) also gave an insignificant increase, whereas the same mixture without superphosphate caused a small decrease in yield.

C. T. Gimingham.

Influence of calcium and nitrogen on the protein metabolism of the soya bean plant. J. M. Ginsburg and J. W. Shive (Soil Sci., 1926, **22**, 175—192).—Soya bean plants grown in limed and unlimed soils, in soil extracts with calcium carbonate, and in complete

culture solutions containing varying concentrations of calcium and nitrogen, were analysed for protein nitrogen, total nitrogen, calcium, and magnesium. There was a definite correlation between calcium in culture solutions and the calcium content of the resultant plants. No definite correlation was observed in the case of nitrogen. Plants showed a higher nitrogen content, however, where calcium carbonate had been added to the medium. Calcium as chloride or nitrate had no influence on the total nitrogen or protein content. High total nitrogen was associated with low hydrogen-ion concentration in the medium. The increased rate of nitrogen absorption in the presence of calcium carbonate had no effect on the protein content. Plants grown with calcium chloride or nitrate required less iron than those grown with calcium carbonate. The plants grown in soil were similar in composition to those grown in culture solutions, but calcium carbonate had a more marked accelerating effect on plant growth in the soil cultures.

G. W. ROBINSON.

Influence of nutrient supply on earliness of maturity in cabbage. J. E. EDMUND and E. P. LEWIS (Michigan Agric. Sta. Tech. Bull., 1925, [75], 1—10; Chem. Abstr., 1926, **20**, 2890).—The greatest response of cabbage plants grown in sand cultures with a modified Pfeiffer's nutrient solution was observed with plants which received applications during the middle growth period; applications during the early growth period caused least response. A surplus of nutrient material, particularly in the middle growth period, increased the length of the internodes. A. A. ELDRIDGE.

Carbon dioxide fertiliser. W. RIEDE (Z. Pflanz. Düng., 1926, **B5**, 383—385; cf. B., 1925, 684).—A "carbon dioxide fertiliser" (58·5% peat, 18% wood charcoal, 3·5% pyrolusite, and 20% nutrient salts) tested under field conditions gave small increases of yield with potatoes and soya beans, but had no effect on cabbages and other market garden crops.

C. T. GIMINGHAM.

Sources of ammonia. J. J. SKINNER and T. S. BUIE (S. Carolina Agric. Exp. Sta. Bull., 1926, [227], 1—32; Chem. Abstr., 1926, **20**, 2890).—New fertiliser materials, obtained from nitrogen-fixation processes, gave favourable results with cotton; with maize the results were good, although sodium nitrate and ammonium sulphate are preferable. A. A. ELDRIDGE.

PATENTS.

"Seed pickling" materials. HOLZVERKOHLUNGS-IND. A.-G. (Austr. P. 100,210, 13.12.21).—Thiocyanates of various metals or mixtures containing the thiocyanate compounds, if necessary with other disinfecting materials, are claimed for use in seed disinfection. For example: (*a*) a solution of barium thiocyanate is added to a solution of aluminium sulphate and the precipitate filtered; (*b*) arsenious oxide and potassium thiocyanate are dissolved in water; (*c*) 10% ferric chloride solution is mixed with 83% of potassium thiocyanate; (*d*) silver nitrate is dissolved in concentrated solutions of potassium or ammonium thiocyanate. For use, such solutions are diluted with water so as to contain, *e.g.*, 0·3% Al, or 0·1% As, or 0·14% Fe, or 0·6% Ag. Other examples are various mixtures of solutions of ferric, magnesium, zinc, mercury, lead, silver, chromium, and copper thiocyanates. Addition of potassium permanganate increases the disinfecting power of the thiocyanates. Addition of phenol, pyridine, or their derivatives is also advantageous. No injury to germination occurs from the use of these solutions. C. T. GIMINGHAM.

Manufacture of cleansing and disinfecting materials, and of fungicides and insecticides. R. VIDAL (F.P. 566,406, 8.8.22; and Addns. 27,591, 30.1.23; 27,779, 14.2.23; 27,784, 19.2.23; 28,059, 10.3.23; 28,060, 15.3.23).—Phenols, or tar oils containing phenols, are converted into alkylated phenols by warming with alcohols in the presence of anhydrous zinc chloride and hydrochloric acid gas. The alkylphenols, *e.g.*, *n*-butyl *o*-cresol, are dissolved in aqueous solutions of alkali and solutions of the alkali soaps of ricinoleic acid are added; or the free phenols are mixed with ordinary soap solutions, castor oil, and aromatic hydrocarbons, carbon disulphide, or tetrahydronaphthalene. The stable emulsions, obtained on dilution with water, find uses in the textile dyeing, tanning, and fur industries, and for disinfection of houses, as well as for destruction of pests of all kinds, in the soil and on plants or animals. Treatment of oleic, palmitic, and stearic acids with alkali hydroxides or ammonia in presence of alkylphenols, phenol, resorcinol, or quinol gives products which, without addition of ricinoleates, can dissolve large amounts of free fatty acids, aliphatic, aromatic, and terpene hydrocarbons, carbon tetrachloride or disulphide. In place of alkylphenols, benzyl-phenols or -cresols, or alkali salts of phenol- or cresol-sulphonic acids, or of *m*-hydroxybenzoic or hydroxytoluic acids can be used. Some of these preparations possess strong lathering properties with sea water or very hard waters. C. T. GIMINGHAM.

Preparation of fungicidal adsorption compounds. CHEM. FABR. VON HEYDEN A.-G., Assees. of D. LAMMERING (G.P. 432,399, 9.7.24).—Soluble copper salts are mixed with alkali aluminates, or only a part of the alkaline reagent required to precipitate the copper is replaced by alkali aluminate. For example, a solution of copper sulphate is poured into a solution of sodium aluminate with stirring; the greater part of the water is removed from the precipitate, which, while still moist, is intimately mixed with fine clay and dried with gentle heating. The product is ground and yields a powder, readily distributable and with good adhesive properties. It is used as a spray fluid in water, or for seed disinfection, or as a fungicidal dust. It remains suspended in water for a long time and adheres specially well to foliage.

C. T. GIMINGHAM.

XVII.—SUGARS; STARCHES; GUMS.

Effect of moisture on the loss of sugar from beets in storage. D. A. PACK (J. Agric. Res., 1926, **32**, 1143—1152).—In order to conserve the maximum amount of sugar in beets, storage must be so controlled that the water content of the roots is kept at its normal, at which point it is under the optimum conditions for growth and respiration. A temperature of about 1·7° inhibits growth and reduces respiration to a minimum. By thus keeping the tissues alive and as dormant as possible, beets may be kept healthy and at a high sugar content for eighteen months.

J. P. OGILVIE.

Dehydration of the sugar beet (De Vecchis process). B. J. OWEN (Intern. Sugar J., 1926, **28**, 542—546).—By using thin beet cossettes, by blowing the air through the sliced material, and by using a temperature not exceeding 110°, it was found possible to dehydrate them to 7½% of water without the formation of invert sugar or caramel. Syrups of 50° Brix were obtained by lixiviation of the dried cossettes. Following treatment with lime and phosphoric acid, a single filtration only was sufficient, but this depended on the quality of the beets. An estimate made of the consumption of steam in manufacture gives 195 lb. of coal per ton of roots worked for ordinary diffusion, but only 50 lb. for the dehydration process on the same basis. It is calculated that by the dehydration process the cost of manufacture would be £5 14s. 5d. per ton of sugar made for a factory slicing 1000 tons of roots per day. J. P. OGILVIE.

Coloration of beet juices during evaporation. V. STANEK and J. VONDRAK (Z. Zuckerind. Czechoslov., 1926, **51**, 1—8, 9—15, 17—22).—An apparatus designed to reproduce in the laboratory the conditions obtaining in the factory in respect of the darkening of juice during evaporation demonstrated, *inter alia*, the following figures: thin-juice, double carbonated, 70—77; thin-juice, carbonated once and sulphited, 19—21; and thin-juice, triple carbonated and unsulphited, 30—34. An increased addition of lime in carbonatation enhanced the resistance to coloration, whilst over-saturation of the first carbonatation gave juices which both before and after evaporation were darker. Regarding the effect of various metals on darkening during evaporation, iron gave the highest figure (124, compared with 102 *in vitro*). J. P. OGILVIE.

Danger of inversion with sulphurous acid in sugar manufacture. L. VAN DER HEIDE (Intern. Sugar J., 1926, **28**, 558—559).—In the sulphitation of cane juice, owing to the presence of buffers, it is possible to carry the operation to a relatively high content of sulphur dioxide without the occurrence of inversion. A p_H less than 7 can be reached provided sufficient buffer salts are present, otherwise there is considerable danger. As a means of obviating this, the addition of some suitable salt, *e.g.*, calcium acetate, in sufficient amount is advised. J. P. OGILVIE.

Formation of caramel substances in the presence of nitrogenous compounds. B. RIPP (Z. Ver. deut. Zuckerind., 1926, 627—655).—Caramel formed by heating a pure solution of lævulose on the water-bath was shown to have a formula identical with that of caramelan (Gelis), possessing four alcoholic hydroxyl groups in the molecule, and behaving as a carbohydrate, *i.e.*, possessing a cupric reducing power and yielding lævulic acid, hydroxymethylfurfuraldehyde, and dextrose on hydrolysis. When the lævulose solution contains nitrogenous substances, the caramel formed no longer behaves in a general way as a carbohydrate, though it still possesses a reducing power. On heating dextrose and lævulose solutions with amino-acids, the depth of colour of the resulting product is due to the interaction with the nitrogenous substance, though not proportionately so. J. P. OGILVIE.

Nature and distribution of the non-sugars affecting the quality of white beet granulated sugars. H. S. PAINE and R. T. BALCH (Intern. Sugar J., 1926, **28**, 472—477).—Particles of impurities large enough to cause considerable turbidity in solutions of white-beet granulated sugars were in some cases found in greatest proportion near the centre of the crystals, indicating that such may act as nuclei. During growth the sugar crystals adsorb continuously colloids of the type which have the property of reducing surface tension and of becoming concentrated at the interface between crystal and liquid, as constituents were found to be distributed through the individual crystals, though present mostly in the external portions, probably owing to the presence of films of mother-liquor on the surface. J. P. OGILVIE.

Determination of sucrose by means of the interferometer. L. HORACEK (Z. Zuckerind. Czechoslov., 1926, **51**, 25—30).—Sucrose may be determined by making two refractometric observations, one before and the other after hydrolysis (preferably using invertase), the increase in the reading being determined by means of the Zeiss interferometer. Although rapid, the method has an accuracy which does not exceed that of the double polarisation process. J. P. OGILVIE.

Fermentation of bagasse. OWEN and BENNETT.—See XVIII.

Determination of sucrose etc. in "stroop." KRUISHEER.—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Brewing in its relations with other fermentation industries. A. FERNBACH (J. Inst. Brew., 1926, **32**, 454—462).—The Nathan process for the manufacture of lager beer supplies one of the most striking examples of the strict application of the principles of pure fermentation, and the methods evolved in its development have been successfully extended, with slight modifications, to the manufacture of vinegar, acetone, butyl alcohol, cider, and wine. The "Amylo" process used in distilleries is the first instance of strictly aseptic work on a large scale in a fermentation industry, and many of its working details are based on brewery methods. The correction of the hydrogen-ion concentrations of worts by the addition of lactic acid ferments has been introduced from distillery procedure into brewing as a means of counteracting alkaline brewing waters. The knowledge of fermentation temperatures acquired in lager beer manufacture and the methods to combat its excessive rise have been applied to distilling for the suppression of the loss of alcohol by evaporation. In wine making, the introduction of refrigeration appliances, suggested by brewing experience, has improved the soundness of the final product. C. RANKEN.

Measurement of foam in beer and the factors which influence it. K. GEYS (Woch. Brau., 1926, **43**, 439—444).—When using the apparatus devised by Lüers for foam measurements decarbonation of the beer due to handling should be avoided. The hydrogen-ion concentration and humulon, with its sensitiveness to the hydrogen-ion concentration, are the two governing

factors in head-retention of beer. Fermentation at low temperatures results in low hydrogen-ion concentrations, whilst the quantity of yeast present has also an effect. Especially with greater quantities of yeast, there is a tendency, towards the end of the fermentation, for the stage of increasing or constant acidity to be followed by a reversion to a more alkaline period. Increasing hydrogen-ion concentration leads to coagulation of the proteins with promotion of foam formation and head-retention, whereas either a constant or a reverting hydrogen-ion concentration causes peptisation of the ultramicrons of the beer, with the result that little foam is formed and the head-retention is poor. This latter result is also assisted by the fact that, with peptisation, the highly charged colloidal particles have no tendency to go to the surface, and, in addition, owing to the extreme smallness of the particles, there are no nuclei at which the carbon dioxide can be evolved. C. RANKEN.

Influence of the brewing water on the acidity of the wort and beer. W. WINDISCH and P. KOLBACH (Woch. Brau., 1926, 43, 423—428, 444—447).—Comparing the effect of the addition of various salts to the brewing water on the worts obtained from the same malt, the authors find that when calcium sulphate is present in the brewing water, the concentration of the hydrogen ions is raised, the amount of buffer substances diminishes owing to the precipitation of calcium phosphate due to the increasing concentration of the calcium ions, but the titration acidity is affected only to a very small extent. Magnesium and sodium carbonates lower the hydrogen-ion concentration, the titration acidity, and also the amount of buffer substances. The loss of buffer substances originates chiefly from the loss in phosphates brought about by the augmented formation of insoluble secondary calcium phosphate by the increased alkalinity. Calcium carbonate diminishes the hydrogen-ion concentration, but to a less extent than does an equivalent amount of magnesium or sodium carbonate. It lessens the titration acidity, and, as the concentration of the calcium ions is increased as is also the alkalinity, these two factors induce a greater precipitation of phosphate with a correspondingly greater decrease of buffering substances. Similar titration curves to those for the worts are found for the beer, but the differences between those obtained for the various salts are very small. No definite conclusions can be reached from the amounts of acidity formed during fermentation, which are deduced from the difference between the titration acidities of the wort and of the corresponding beer. In the majority of cases, the amount of buffer substances in the beer is less than that in the wort, but in a few cases the converse holds. C. RANKEN.

Influence of temperature on the optimal hydrogen-ion concentration for amylolytic action. H. LÜERS and S. NISHIMURA (Woch. Brau., 1926, 43, 415—416).—The results of the authors are not in concordance with those of Olsen and Fine (B., 1924, 992), who had previously showed that the optimal p_H for the amylolytic action of malts varied from 4·3 to 6·0 for the temperature range of 25—70°. Considering that the experimental conditions of the previous workers were not sufficiently definite and controlled, they have repeated the investigation, using a solution of soluble starch, which, after being buffered by acetate-acetic acid mixtures to give p_H values varying from 3·5 to 6·0, was fermented by an extremely active form of amylase prepared according to the method of Sherman and Schlesinger. For 15—70° the optimal p_H was found to lie within the limits p_H 4·4 to 4·6, and the zone of optimal action became more restricted with rising temperature. C. RANKEN.

Determination of the colour of wort and beer by colorimeter constructed according to Ostwald's theory. F. MESTAN (Z. angew. chem., 1926, **39**, 1336—1340, and Chem. Listy, 1926, **20**, 462—468).—The author discusses the defects of the different instruments for the measurement of colour on the basis of Ostwald's principles (B., 1919, 914 A), and describes an improved form of apparatus which is based on the use of wedges of coloured glass, the rays transmitted through these wedges being compared with those passing through the liquid under examination. C. RANKEN.

Fermentation of bagasse in relation to the yield of industrial alcohol. W. L. OWEN and N. BENNETT (Intern. Sugar J., 1926, **28**, 463—469).—Addition of bagasse to molasses worts decreases the efficiency of the fermentation (making allowance for the sugars introduced), this probably being due to an inhibiting effect of gums, non-fermenting sugars, and other substances present. J. P. OGILVIE.

Determination of tartaric acid in wine. DUBOUX.—See XXIII.

PATENTS.

Obtaining acetone and other products by fermentation of materials containing carbohydrates. N. MOSKOVITS (Austr. P. 102,927, 6.3.22).—The materials are treated in closed vessels with organisms which generate acetone. The fermentation is carried out in the same vessel in which the steaming of the raw materials and the preparation of the mash are performed. The vessel is connected with a gasholder containing, under pressure, a gas (*e.g.*, air, oxygen, nitrogen, or ammonia) which does not hinder fermentation so that, when the connexion is opened after steaming, the gas enters as the pressure in the fermentation vessel falls on cooling. The entrance of foreign organisms before and during fermentation is thus prevented. In other respects the process follows the usual method for obtaining acetone by fermentation of materials containing carbohydrates, with, *e.g.*, *B. macerans*. Indifferent substances such as asbestos, filter-paper, beer refuse, potato skins, straw, and nitrogenous organic materials, like compressed yeast, yeast extract, or malt embryos, may be added to the mash, and, in sterilising the mash, acids may be added. A. DAVIDSON.

XIX.—FOODS.

Refractive index and density of butter fat. A. SCHNECK (Milchw. Zentr., 1926, **55**, 113—116, 153—156).—The values of the specific refraction, obtained from the refractive index and density by the Lorentz-Lorenz formula, of a number of butter fats show only slight variations among themselves, and approximate closely to the theoretical values calculated from the percentages of esters and the refractivities of their constituent atoms.

Assuming a mean specific refraction, the density of a butter fat may be calculated from its refractive index and *vice versa*, the results differing from the observed values by no more than one or two units in the third decimal figure. Summer butter has a higher specific refraction than winter butter; also butter from the later stages shows higher values than that from the earlier stages of lactation. F. R. Ennos.

Determination of flour in sausages. H. S. J. F. Snethlage (Chem. Weekblad, 1926, **23**, 465—466).—As the extract with 25% hydrochloric acid recommended for the determination by Dutch law is found to give rapidly diminishing rotations on keeping for a few hours, it is recommended that the extract be diluted to contain 15% of acid, before taking the polarimeter reading. Direct extraction with 15% acid is not satisfactory. S. I. Levy.

Determination of sucrose and invert sugar, and of glucose (starch) syrup in "stroop." C. I. Kruisheer (Chem. Weekblad, 1926, **23**, 430—437).—In order to arrive at a suitable method of analysis for the syrup commonly used as a sugar foodstuff in Holland under the name "stroop," and which consists of a mixture of glucose (starch) syrup and molasses, samples of these component materials from the best-known Dutch makers of the material were analysed separately, and mixtures in various proportions were then prepared and tested. The method of analysis adopted for the mixtures, which is accurate to within about 2%, consists in warming with dilute acid to cause inversion, making alkaline, adding excess of iodine, removing the excess exactly, and determining the reducing sugars expressed as lævulose by Schoorl's method (cf. Kolthoff, B., 1923, 467A); the figure obtained × 1·9 gives the sucrose and invert sugar together expressed as sucrose. For determination of the starch syrup the sample is hydrolysed by boiling for 1 hr. with hydrochloric acid, and the reducing sugars are determined as before. Formulæ are given for expressing the composition of "stroop" from these determinations. S. I. Levy.

Composition of the food grains, vegetables, and fruits of Western India. D. L. Sahasrabuddhe (Dept. Agric. Bombay Presidency Bull., 1925, [124], 1—38; Chem. Abstr., 1926, **20**, 3049).

Patent.

Making edible alkali caseinate. H. F. Zoller (U.S.P. 1,598,334, 31.8.26. Appl., 8.5.22).—Sufficient alkali solution to dissolve the casein is added with stirring to a suspension, in a dilute alkali phosphate solution, of casein which has been precipitated by acid, the use of considerable excess of alkali being avoided. L. A. Coles.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Production of ether by solid catylysts. S. K. K. Jatkar and H. E. Watson (J. Indian Inst. Sci., 1926, **9A**, 71—109).—A detailed account of results previously published (J.S.C.I., 1926, **45**, 168 T).—Measurements of the equilibrium constant made by determining the quantity of alcohol formed when mixtures of ether and water are passed over the alum catalyst indicated that the most probable value is 7·3 at 500° Abs. E. H. Sharples.

Crystallisation of citric acid from lemon juice. G. Ajon (Riv. Ital. Essenze Profumi, 1925, **7**, 125—127; Chem. Zentr., 1926, II, 1597—1598).—The juice, which is prevented from coming in contact with iron by the use of porcelain presses and basins, is stored for several days, preferably with the addition of some fermenting juice, when it is concentrated to one-fourth of its volume, and inorganic constituents are precipitated and removed. After further concentration on the water bath, and the addition of a little sulphuric acid, citric acid separates in fine crystals. Large crystals are obtained by subjecting the juice to osmosis after removing the inorganic constituents (cf. B., 1925, 227; 1926, 214). L. A. Coles.

Analysis of commercial lactic acid. U. J. Thuau and M. Vidal (J. Soc. Leather Trades Chem., 1926, **10**, 257—258).—Sulphuric acid can be determined in lactic acid by the Meunier method, in which the sulphates are precipitated by means of 95% alcohol, the latter evaporated off from the filtrate, and the sulphuric acid determined by means of barium chloride. In the Balland method, the total SO_4 is determined by means of barium chloride, the combined SO_4 by determining the sulphates in the ash, and the difference is due to free sulphuric acid. In both methods the result should be converted into the equivalent of lactic acid and deducted from the percentage of the latter as determined by Thompson's method (cf. B., 1918, 343 A). To determine hydrochloric acid, a weighed amount of the sample is neutralised with alkali, evaporated to dryness, heated carefully without calcining, extracted with hot water, and the total chlorides determined (I). The determination is repeated, omitting the neutralisation (II). The difference (I—II) gives the chlorides due to free hydrochloric acid. A sample of lactic acid analysed by the authors contained 50·70% of lactic acid as determined by the official method, but 2·27% of this was due to free sulphuric acid, so that the real content of lactic acid was only 48·43%. D. Woodroffe.

Stability of hexylresorcinol in pharmaceutical preparations. W. A. Feirer and V. Leonard (J. Pharm. Exp. Ther., 1926, **28**, 395—397).—Hexylresorcinol, in solution in olive oil enclosed in soluble gelatin capsules, does not deteriorate on keeping for one year at the ordinary temperature. H. I. Coombs.

Terpineol content of turpentine obtained in the production of terpin hydrate. Susskind (Trans. Sci. Chem. Pharm. Inst. Moscow, 1923, [26], 29—30; Chem. Abstr., 1926, **20**, 3076).—The mother-liquor from the preparation of terpin hydrate from turpentine and sulphuric acid, when distilled in steam, yields terpineol (8—9%) and pinene (38%). A. A. Eldridge.

Automatic devices for extracting alkaloidal solutions. II. Application to nux vomica and belladonna alkaloids. H. R. Watkins and S. Palkin (J. Amer. Pharm. Assoc., 1925, **14**, 1099—1104; Chem. Abstr., 1926, **20**, 2895).—Automatic extractors are

preferable to hand-shaken separators. The quantity of ammonia or ammonium sulphate present is immaterial when chloroform or benzene is used; small losses of atropine were observed when ethyl ether was employed in the presence of ammonia. A. A. ELDRIDGE.

Iodine value of commercial peptones. A. BERTHELOT and M. CNADUC (Bull. Soc. Chim. biol., 1926, **8**, 936—939).—The iodine value (determined titrimetrically) in acid and in alkaline solution is suggested as a useful method for the comparison of commercial peptones for bacteriological purposes. R. K. CANNAN.

Activity of the commercial extracts of parathyroid glands. M. KOHLER (Biochem. Z., 1926, **175**, 27—30).—Extracts prepared by Collip's method (A., 1925, i, 754, 1017) gave often a transient fall followed by a considerable rise, gradually attained, in blood calcium. Of four commercial extracts two had no effect, one caused a slight fall, and the fourth a slight rise in blood calcium. Hæmocrinine, a dry blood preparation containing the hormone, also was inactive. P. W. CLUTTERBUCK.

Volatile oil of *Collinsonia anisata*. E. R. MILLER and J. K. HUNT (J. Amer. Pharm. Assoc., 1925, **14**, 1096—1098; Chem. Abstr., 1926, **20**, 2895).—The oil (yield 0·138%) had α_D $-2·34°$ to $-0·4°$, $n_D^{19.2}$ 1·5185—1·5225; about 80% is methylchavicol. Salicylic acid, safrole, and an unidentified ketone, terpene, and aldehyde are also present. A. A. ELDRIDGE.

Volatile oil of *Myrica asplenifolia*, Endl. M. A. BRAUN (J. Amer. Pharm. Assoc., 1926, **15**, 336—337; Chem. Abstr., 1926, **20**, 2896).—The oil (yield 0·02%) had d^{25} 0·8945, α_D $-3·75°$. Aldehyde was absent from the lowest fraction (below 100°) but present in the third (150—200°). The 200—220° fraction had an ester content of 28·8%, whilst the total alcohol content was 45·74%. A. A. ELDRIDGE.

PATENTS.

Recovery of alcohol, ether, aldehydes, etc. from gases. E. BERL (G.P. 432,357, 3.3.22).—Organic vapours which are present in dilute form in air, hydrogen, or waste fumes are recovered by absorption in relatively small quantities of coal tar phenols or lignite tar phenols, any residual vapour being adsorbed by activated charcoal or colloidal silica. W. G. CAREY.

Manufacture of phosphoric esters of polyhydric alcohols. P. E. GOISSEDET and A. L. HUSSON, Assrs. to SOC. CHIM. DES USINES DU RHÔNE (U.S.P. 1,598,370, 31.8.26. Appl., 15.9.25. Conv., 27.4.25).—Polyhydric alcohols are treated with phosphoric anhydride in the presence of tertiary bases. The esters are precipitated as their calcium salts. W. N. HOYTE.

Manufacture of primary and secondary aromatic amines. E. MERCK, Assees. of W. KRAUSS (G.P. 432,151, 21.8.24; Addn. to 407,487, B., 1925, 474).—The chief patent and G.P. 417,926 (B., 1926, 173) are varied by treatment of secondary or tertiary amines of the general formula Aryl·NRR′ (R = H, alkyl, or benzyl; R′ = benzyl or substituted benzyl) with hydrogen in presence of metal catalysts. For example, ethylbenzylaniline and alcohol are shaken with hydrogen in presence of palladium black. Exactly 1 mol. of hydrogen is absorbed yielding toluene and ethylaniline. The following amines are decomposed in a similar manner:—methylbenzylaniline into methylaniline and toluene; benzylaniline into aniline and toluene; dibenzylaniline into aniline and toluene; 3 : 4-methylenedioxybenzylaniline into aniline and 3 : 4-methylenedioxytoluene; *o*-hydroxybenzylaniline into *o*-cresol and aniline. A. DAVIDSON.

Preparation of di- and poly-sulphochlorides of homo- and hetero-cyclic mono- and poly-nuclear aromatic compounds and their substitution products. O. LUSTIG and E. KATSCHER (Austr. P. 101,667, 9.5.23).—The starting material is heated with an excess of chlorosulphonic acid (at least 5 times the weight) for a long time (5 hrs. at least) at the b.p. of the reaction mixture or just below it. Examples are given of the preparation of m-*xylenedisulphonyl chloride* from xylene, of 1-*chlorobenzene*-2 : 4-*disulphonyl chloride* from chlorobenzene, and of *carbazoletetrasulphonyl chloride* from potassium carbazolesulphonate. Yields of 75—80% of crystalline products are obtained. A. DAVIDSON.

Preparation of carboxylic esters of halogenated polyhydric alcohols. S. ROSENZWEIG and H. LEGERLOTZ (Austr. P. 101,671, 9.11.23).—A mixture of a polyhydric alcohol and a carboxylic acid is treated with dry hydrogen halide at a suitable temperature. For example, dry hydrogen chloride is passed into a mixture of *p*-acetamidobenzoic acid and glycol at 110—120° for several hours, and the glycolchlorohydrin produced is distilled off. On pouring the residue into an excess of sodium carbonate solution, β-*chloroethyl* p-*aminobenzoate*, m.p. 85—86°, crystallises out. Similarly from *p*-nitrobenzoic acid, glycol, and hydrogen chloride at 100—110° is formed β-*chloroethyl* p-*nitrobenzoate*, m.p. 54—55°. By passing dry hydrogen bromide into a mixture of equal parts of anhydrous glycerol and acetic acid at 140—160°, the ester produced being simultaneously distilled off, treated with dilute sodium carbonate, dried with calcium chloride, and distilled in a vacuum, *monobromohydrin acetate*, $CH_2Br·CH(OH)·CH_2·O·COMe$, b.p. 172—174°/100 mm., is obtained. A. DAVIDSON.

Manufacture of an acid crystalline saponin from *Primula* species. L. KOPLER and S. ROSENZWEIG (Austr. P. 101,486, 21.3.24).—Primula plants are extracted with aqueous alkaline solutions, and the saponin is precipitated from the extracts by acidification and filtered off. The crude saponin is dissolved in hot dilute alcohol, the solution treated with decolorising charcoal, filtered, and after distilling off most of the alcohol, the saponin is precipitated by gradual addition of water and crystallised from suitable solvents. Alternatively, the plants are extracted with hot dilute alcohol and, after distilling off most of the alcohol, the saponin is precipitated by water, filtered off, again dissolved in hot dilute alcohol, and purified as above. The end products or intermediate products of this process are subjected to electrodialysis in alkaline aqueous solution. A. DAVIDSON.

Alkylhydroxyalkyl- and dihydroxyalkyl-arsinic acids. ÉTAB. POULENC FRÈRES, and C. J. OECHSLIN (F.P. 585,970, 15.11.23; cf. E.P. 206,152, B., 1925, 83).—Alkyl or hydroxyalkyl halides are allowed to act on sodium salts of hydroxyalkyl- or alkyl-arsinic acids. For

example, an aqueous solution of sodium methylarsinate is reduced at 45° with sulphur dioxide and neutralised with sodium hydroxide solution (d 1·332). Glycolchlorohydrin is added to the solution at 30°, the mixture stirred 6—8 hrs., acidified with sulphuric acid, evaporated in a vacuum, the residue extracted with hot alcohol, and the alcoholic solution allowed to crystallise or precipitated with acetone or ether. The *methylhydroxyethylarsinic acid*, $OH\cdot CH_2\cdot CH_2\cdot AsMe\cdot O_2H$, softening between 80° and 103°, forms water-soluble salts. The same acid is obtained from sodium hydroxyethylarsinate and methyl iodide. By the action of glycolbromohydrin on sodium hydroxyethylarsinate, *dihydroxyethylarsinic acid* is formed. From sodium hydroxyethylarsinate by treatment with allyl bromide at 60°, *allylhydroxyethylarsinic acid* is formed. A. DAVIDSON.

Manufacture of mercurated hydroaromatic hydrocarbons. F. NEUMANN (Austr. P. 100,723, 14.11.23).—Mono- or poly-cyclic aromatic compounds, fully or partly hydrogenated, are treated with mercuric salts at about 150—250°. For example, tetrahydronaphthalene is heated at about 200° for ½ hr. with less than 1 mol. of mercuric acetate; the *mercurated tetrahydronaphthalene* forms needles of no definite melting point and decomposes slowly on heating. The preparation of analogous *compounds* from decahydronaphthalene and *cyclo*hexanone is described. The products are soluble in indifferent organic solvents such as carbon disulphide, are strongly toxic, and find application as disinfectants and as a means of combating plant pests. A. DAVIDSON.

Preparation of highly purified physiologically active substances from female internal secretive organs. GES. FÜR CHEM. IND. IN BASEL (Swiss P. 113,835, 5.12.24).—Fatty acids and other acid impurities may be removed from extracts of organs by treatment with alkalis or alkaline earths, the extracts being dissolved in solvents in which the resultant soaps etc. are insoluble; or the alkalis etc. may be dispersed on an indifferent carrier (carbon, kieselguhr, etc.), the treatment being carried out as before, or by impregnating the loaded carrier with the extract and extracting with a solvent in which the resultant soap etc. is insoluble. The alkalis etc. may be dispersed by, *e.g.*, treating animal charcoal with an aqueous alcoholic solution of sodium hydroxide or sodium carbonate and evaporating. Examples are given of the purification of extracts of placenta, ovary, and corpus luteum. B. FULLMAN.

Preparation of a remedy for diabetes. J. BURMANN (Swiss P. 107,092, 23.5.23).—Fresh glands with internal or external secretion, such as pancreas or liver, from healthy animals are freed from tissue and fat, treated with physiological serum and 1% of their weight of hydrochloric acid, and ground in an indifferent gaseous atmosphere. Sterile sand is added, the mass ground in absence of air at 0°, and filter-pressed. The resultant liquid contains all the soluble enzymes (especially the glycolytic ones) as well as the hormones and hydrochlorides of the active bases of the glands. It is dried, and the residue, after removal of fat by treatment with light petroleum, dissolved in water and the solution added to ether, when a fine powder is precipitated. This has a strong diastatic action, and gives good results in the treatment of diabetes. The same product is obtained from yeast of the species *Saccharomyces ellipsoideus glykolyticus*. The yeast is cultivated in a medium containing glycerophosphoric acid. On completion of the fermentation, the yeast is allowed to settle, washed with water faintly acidified with hydrofluoric acid, ground, and then treated as above. B. FULLMAN.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Ripening of photographic silver halide emulsions. W. JENISCH (Z. wiss. Phot., 1926, 24, 248—256).—Lüppo-Cramer's experimental proof of the nucleus theory of sensitivity depends on the observation of the removal of the nuclei with chromic acid and the resultant decrease in sensitivity. Conversely, it follows that the addition of nuclei should result in an increase of sensitivity. The observations of Lüppo-Cramer and others, that colloidal metal sols added to a finished emulsion before coating are without influence on speed and fog, were confirmed. Colloidal gold, present in an acid emulsion during precipitation, gave a marked increase of sensitivity with no fog. Increase in the amount of gold gives a bigger speed increase and, eventually, fog. Ammonia-emulsions behave similarly. The influence of the colloidal metal depends on its dispersity, the sensitising action increasing with increasing dispersity, and the amount of metal sol that can be added without inducing fog increases also. In the case of a fine gold sol prepared by phosphorus reduction it was possible to add a certain amount of sol without producing any effect. Silver and platinum sols give increased sensitivity to about equal extents, but not so markedly as the highly dispersed gold sols. The results establish that part of the sensitivity increase in ripening is due to formation of nuclei in the last part of the ripening process, but the nuclei are not necessarily of silver. W. CLARK.

Silver chloride grain. R. E. LIESEGANG (Phot. Ind., 1926, 867—868).—Sodium chloride spheres, 5 mm. in diameter, were converted into silver chloride spheres by immersing for long periods in concentrated silver nitrate solution, and these were used to imitate the microscopic grain of the photographic plate. If such spheres are exposed and developed in metol–adurol, reduction occurs at the surface with formation of grey-white metallic silver, and the shape is unaltered. Oxidation of the developer is more rapid in the neighbourhood of the grains than at the surface of the solution by the air. A model of a photographic plate was prepared by immersing a silver chloride sphere in a gelatin sol, and setting. When a developer was poured on the jelly surface, it became dark brown when it had diffused to the sphere. If, after development, the gelatin was melted, the sphere remained surrounded by an envelope of tanned gelatin. Sulphide toning may be illustrated by treating the developed sphere with sodium sulphide solution. The solvent action of thiosulphate and ammonia may also be observed; the action begins at some particular point on the sphere, and is not regular. W. CLARK.

Desensitisation. LÜPPO-CRAMER (Phot. Ind., 1926, 948—952).—The paper deals essentially with the statement of Lumière and Seyewetz that phenosafranine can be completely washed out of a plate, with restoration of the original sensitivity, and the conclusion that this indicates that desensitisation is not an oxidation effect, but is due to the formation of a less light-sensitive complex between the desensitiser and the silver bromide. Desensitisation is certainly decreased by washing, but it is not completely destroyed, the extent of the decrease varying with the desensitiser. If the statement of Lumière and Seyewetz were correct it would not disprove the oxidation theory of desensitisation. The results of Hübl (B., 1926, 612) are quoted in support of the arguments. W. CLARK.

PATENTS.

Film for photocollographic printing plates. M. DE'SPERATI, Assr. to ARGENTOGRAPHICA, LTD. (U.S.P. 1,598,061, 31.8.26. Appl., 28.11.25).—A film for photocollographic printing plates has a gelatin layer on each face. One of the layers is prepared so as to dissolve at a lower temperature than the gelatin of the other layer. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Liquid air explosives. LIQUID OXYGEN EXPLOSIVES, LTD. From LES PETITS-FILS DE F. DE WENDEL ET CIE. (E.P. 257,828, 26.2.26).—A liquid explosive charge contains cooling salts such as sodium chloride or sodium carbonate, and blast-furnace dust as an absorbent material for the liquid air. S. BINNING.

Detonator [priming composition]. E. VON HERZ (E.P. 241,892, 15.10.25. Conv., 23.10.24).—Metal salts of the *iso*nitroamines are used in priming compositions for detonators and percussion caps. The lead salts of methylene di*iso*nitroamine and ethylidene di*iso*nitroamine are specially mentioned. They may be used in detonators in conjunction with lead styphnate. A mixed salt obtained by the simultaneous precipitation of the lead salts of an *iso*nitroamine and of styphnic acid is very effective as a priming composition. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Water purification studies by U.S.A. Public Health Service. H. W. STREETER (J. Amer. Water Works Assoc., 1926, 16, 336—341).—A preliminary review of experimental work completed and in progress. The results obtained in a collective survey of a group of 17 municipal filtration plants made in 1923—4 to determine the maximum limits of raw water pollution which are consistent with the production of effluents meeting given standards of bacterial purity have been confirmed by the experimental work in which a small but fully equipped rapid sand filter plant was used. The results indicate that the maximum *B. coli* index of the raw water consistent with the production of chlorinated filter effluents conforming to the revised Treasury Department standard is approximately 5000—6000 per 100 c.c., and as regards unchlorinated effluents 60—100 *B. coli* per 100 c.c. Although overall efficiency of bacterial removal, as measured in terms of 37° plate count, is influenced decidedly by turbidity and seasonal changes, the corresponding overall efficiency of removal of organisms of the *B. coli* group appears to be affected only to a minor, if any, extent by either of these factors; and in general, the overall efficiency of bacterial removal, and more especially of *B. coli* removal, is affected to a considerably greater extent by variations in the density of bacteria in the raw water. W. T. LOCKETT.

Sand filtration. M. E. DICE (Chem. Met. Eng., 1926, **33**, 529—533).—Air injected during the softening of water becomes entrained in the water and penetrates into the sand of gravity filters, thus making a porous structure, and allowing the precipitate to work its way into the sand instead of forming a film on the surface. The air can be removed by a vacuum process or by raising the pressure sufficiently to dissolve the air, for example, by increasing the hydrostatic head in the filter tank, but this procedure is not advisable where the removal of bacteria is desired. No penetration of air into the sand bed occurred with a head of 12·3 ft. or more of water. An empirical formula for the rate of flow of water through sand was checked experimentally and was accurate for clear water, and a formula was evolved for determining the rate of filtration at any time for the particular sediment-bearing water examined. Sand for filtration should have a low "coefficient of uniformity," this being the ratio of the sieve opening which will pass 60% of the sand to that which will pass 10%. The sand also should have a low "effective diameter," *i.e.*, the screen opening, expressed in millimetres, which will allow 90% of the sand to pass through. W. G. CAREY.

Physico-chemical analyses by seeded precipitation: applications to the determination of lime in waters and tartaric acid in wine. M. DUBOUX (Ann. Chim. analyt., 1926, **8**, 257—264).—The electrometric titration of substances by a precipitating agent is a slow process in dilute solutions ($N/300$ to $N/3000$) owing to the time taken for the precipitate to form. By "seeding" the solution with the appropriate precipitate—which has been previously prepared by the usual analytical procedure, thoroughly washed, and preserved in the moist condition—the time required for titration is considerably curtailed. In potable waters calcium is determined, after decomposition of bicarbonates, by electrometric titration with sodium ammonium racemate, which does not precipitate magnesium, the solution being seeded with calcium racemate. For the determination of *d*-tartaric acid in wine, an excess of ammonium *l*-tartrate is added and the solution titrated with calcium acetate after the addition of calcium racemate. A correction factor must be applied in this case owing to the entanglement of calcium *l*-tartrate with the precipitated racemate. The results compare favourably with those obtained by the bitartrate method. F. R. ENNOS.

Determination of cresols. DANCKWORTT and SIEBLER.—See III.

Treatment of waste tannery waters. NOYER.—See XV.

PATENT.

[Apparatus for] preserving sea-water for biological purposes. J. KUNSTLER (E.P. 241,893, 16.10.25. Conv., 25.10.24).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DECEMBER 10, 1926.

I.—GENERAL; PLANT; MACHINERY.

Determination of the filtration constants of various charcoals. H. I. Waterman and A. Dauvillier (Rec. trav. chim., 1926, **45**, 628—632; cf. *ibid.*, 1924, **43**, 757).—The constants m and n of the filtration formula $dV/d\theta = K_1P^n/V^m$, where V is the volume of liquid filtering in time θ, P the pressure, and K_1 a constant depending on apparatus and temperature, have been determined for carboraffin, norit, and nobrac by measuring the speed of filtration of suspensions of the charcoals in water under different pressures. The values of m and n are only approximately constant, their order of magnitude being: carboraffin $m = 0 \cdot 62$, $n = 0 \cdot 56$; norit $m = 0 \cdot 63$, $n = 0 \cdot 72$; nobrac $m = 0 \cdot 36$, $n = -0 \cdot 30$. The negative value of n for the nobrac shows that the speed of filtration decreases with increase of pressure, probably owing to the greater compressibility of the nobrac. The charcoal layer obtained on the filter cloth was quite air-tight though not water-tight, and was only removed by very thorough washing.

S. J. Gregg.

Gas apparatus. G. Stiehr (Chem.-Ztg., 1926, **50**, 694).—A pocket apparatus for the detection and determination of small quantities of substances, *e.g.*, calcium carbonate in soil, which yield gases on treatment with a suitable liquid. The gas evolved is measured by counting the bubbles of air which are displaced through water and standardising the apparatus with weighed amounts of known substances.

F. R. Ennos.

[Laboratory] filtering devices. H. B. Gordon (Ind. Eng. Chem., 1926, **18**, 1075—1076).—Filtration of a large volume of liquid, without the necessity of constant attention, can be effected by a device in which the liquid is contained in a carboy with its stopper perforated by two tubes. One is a siphon leading to the filter, the other an air tube terminating within the filter funnel but near the top. When the funnel fills, the liquid seals this air tube and stops the flow until the level has fallen. If it is necessary that the liquid should be in a wide-mouthed vessel a siphon tube only is used, which, together with the funnel, is supported by a clamp and occasionally lowered as the vessel empties.

C. Irwin.

Apparatus for examining oils. Kiemstedt.—See II.

Counter-current jet condenser. Müller.—See II.

Apparatus for measuring turbidity and colour of water. Olszewski.—See XXIII.

Patents.

Performing chemical reactions. E. C. R. Marks. From A.-G. f. Chemiewerte (E.P. 231,901, 6.4.25).—The use of a reactive agent comprising a porous carrier moistened and treated with a reacting substance under reduced pressure facilitates and intensifies chemical reactions. Thus, by impregnating kieselguhr or fuller's earth under reduced pressure with sulphuric acid, and removing the excess by pressing, the remaining water being then eliminated by heating the residue *in vacuo*, small quantities of active substances remain finely distributed in the pores. Various applications are suggested.

F. G. Crosse.

Pulverising apparatus. J. E. Bell and W. J. A. London, Assrs. to Int. Combustion Eng. Corp. (U.S.P. 1,598,702, 7.9.26. Appl., 8.1.24).—A centrifugal machine is described having an impeller disc with detachable impeller blades, together with an annular receiving chamber and impact blades interposed between the disc and chamber.

F. G. Crosse.

Generation of steam and other vapours. Niederbayerische Cellulose-werke A.-G., and A. Schneider (E.P. 253,062, 11.8.25. Conv., 6.6.25).—In a Field boiler tube unit, by throttling the steam outlet and providing a non-return valve in the water or down tube, the excess pressure in the up or steam tube will cause it to remain partially empty of water and the upper part will serve as a superheater, the steam being further dried on expansion through the throttling device into the boiler.

B. M. Venables.

Heat interchanging apparatus. L. Chavanne (E.P. 249,535, 19.3.25. Conv., 19.3.24).—The apparatus comprises a nest of tubes, together with the necessary diaphragms to form headers and baffles in a casing, the whole being made into a unit of box-like form. The flow of the internal fluid is in one direction through half the tubes and back through the other half. The external fluid also passes twice along the tubes. Refractory material may be placed either inside or outside the tubes to aid the heat interchanges.

B. M. Venables.

Condenser type heaters. Griscom-Russell Co., Assees. of J. Price (E.P. 249,079, 28.12.25. Conv., 13.3.25).—A heat interchanger comprising a nest of tubes is provided with one or more supporting diaphragms for the tubes, the whole unit being easily removable from the shell. A baffle for deflecting gases to the appropriate discharge outlet may also be combined with the tube bundle.

B. M. Venables.

Drying, heating, or cooling machines. W. G. and F. R. Simon (E.P. 258,978, 23.7.25).—An apparatus comprising a bundle of heating or cooling tubes rotating in a trough-like chamber, allowing vapours to be drawn off from the chamber at positions other than and in addition to the usual ones at the ends.

B. M. Venables.

Centrifugal separators for liquids. F. GRIMBLE, M. N. CAIRD, and E. COOMBS (E.P. 258,097, 12.10.25).—A centrifugal separator provided with separate outlets for both liquids, is provided with a device for closing the outlet for heavier liquid when the separator is used for clarification purposes, *i.e.*, when the quantity of heavier constituent is so small that the light constituent might overflow at the wrong place. The heavier liquid normally flows over a weir formed on the rim of the bowl; this outlet may be closed by a conical ring.
B. M. VENABLES.

Centrifugal separators. B. C. CARTER (E.P. 258,204, 14.3.25).—Centrifugal separators for removing impurities from fluids, *e.g.*, from the oil and air used in internal-combustion engines, are constructed so that the fluid passes directly through the apparatus in an axial direction in passages at a short distance from the axis between an inner perforated rotating wall and the shaft. The impurities are subjected to greater centrifugal force (*i.e.*, at a larger radius) after passing through the inner wall, and may be caught on an outer peripheral rotating wall, which in the case of lubricating oil being cleaned would not be perforated, but perforations are necessary in the case of air to allow the dust to be continually discharged. For liquids such as oil an air outlet practically coincident with the axis is provided. The passage of the fluid through the apparatus is sufficient to drive turbine blades which cause the apparatus to rotate.
B. M. VENABLES.

Centrifugal separators. G. FRANCIS (E.P. 258,629, 23.3.25).—In a separator yielding two liquid products the liquid under treatment is caused to flow in a sinuous course while in the separating bowl; to this end discs (or cones) are provided with openings near the circumference and near the axis in alternate discs, and either the inner or outer edges of the discs are sealed by either a solid or a liquid seal. The liquid, if used, is of different specific gravity from the liquid under treatment.
B. M. VENABLES.

Evaporators for refrigerating apparatus. S. OTTO, Assee. of J. C. JANKUS (E.P. 245,124, 19.12.25. Conv., 19.12.24).—In a reversing absorption refrigerator any liquid absorbent (water) which may collect in the evaporator or refrigerator proper passes to a sump below through a restricted orifice. From the sump communication is provided to a receiver for liquefied refrigerant above, so that the liquid will be drawn up when the pressure drops.
B. M. VENABLES.

Evaporator plant. J. MUGLER (U.S.P. 1,598,301, 31.8.26. Appl., 8.2.26).—The ill-effects of varying steam pressure in an evaporator plant are prevented by providing the regular evaporators with an additional unit which operates either as an evaporator or as a discharge vessel for the liquid under treatment. Overflow pipes for the vapours connecting the units are equipped with automatic regulators so that steam of higher pressure may be supplied from one unit to the next. In this way the efficiency of the plant may be kept constant irrespective of any variations in the steam consumption.
R. B. CLARKE.

Separation of the lowest boiling constituents of a mixture of liquids by vacuum distillation. A. SCHMALENBACH (E.P. 244,736, 23.11.25. Conv., 22.12.24).—In vacuum distillation the vapour of the condensate is liable to reduce the vacuum attainable. The gases and vapours going to the vacuum pump are, therefore, scrubbed with another liquid which removes them, and the vapour is recovered in a rectifying column which operates under a lower vacuum. The same vacuum pump may be used for both stages by inserting in the second stage a vacuum reducer comprising a deep liquid seal.
B. M. VENABLES.

Apparatus for evaporating, distilling, or concentrating liquid. SOC. DES CONDENSEURS DELAS (E.P. 258,165, 27.2.26. Conv., 18.1.26).—In a vapour compression evaporator where the compression is effected by a live steam ejector the diffuser or outlet nozzle is cooled by the incoming feed liquid; by this means the efficiency of the ejector considered as a pump is increased, and the heat abstracted is not lost. Additional preheaters may be provided for the feed liquid after it has passed through the diffuser jacket.
B. M. VENABLES.

Liquefaction and separation of gaseous mixtures. R. C. E. MEWES (E.P. 258,773, 25.5.25).—The constituents of a gas mixture (*e.g.*, air) are separated by partial condensation at moderate pressure followed by rectification. The condenser is cooled by a separate portion of air compressed to 150—200 atm. and expanded to 50 atm., the liquefied portion being expanded to 3—6 atm. before entering the condenser for the main portion of air. The gas not liquefied at 50 atm. is returned to the compressor and raised to 150—200 atm. again. The condensate (rich in oxygen) is rectified, and liquid low-boiling gas (rich in nitrogen) is used as the heating agent. By this means the products may be removed under reduced pressure, and the condensate from the rectifier sent to the liquefier. B. M. VENABLES.

Tintometer. BRIT. LAUNDERERS' RESEARCH ASSOC. and R. G. PARKER (E.P. 258,366, 25.6.25).—A number of discs of plaster of Paris, tinted by mixing various pigments with the dry plaster before being cast in moulds, are compared with the surface to be examined through a black screen provided with two holes. The standard discs are changed until one is obviously lighter and another obviously darker than the surface under observation.
B. M. VENABLES.

Measuring the amount of gas dissolved in a liquid. H. A. DAYNES, and CAMBRIDGE INSTRUMENT CO., LTD. (E.P. 259,086, 22.1.26).—The amount of gas dissolved in a liquid, more particularly oxygen in water, is measured by scrubbing a second gas (hydrogen) with the liquid, by which means the oxygen will be removed from solution, and comparing the heat conductivity of the hydrogen–oxygen mixture with that of the original hydrogen in a catharometer. (See E.P. 124,453; B., 1919, 393 A.)
B. M. VENABLES.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Determination of volatile matter in solid fuels. D. J. W. KREULEN (Chem. Weekblad, 1926, **23**, 477—479).—The method proposed by de Waard (cf. B., 1925, 979) is criticised as unsuitable, differences of 2·12% having been found.
S. I. LEVY.

Rapid determination of sulphur in coal by the method of Bahr and von der Heide. W. Melzer (Brennstoff-Chem., 1926, 7, 313—314; cf. B., 1925, 33).—Errors in the method, varying in amount with different coals, are introduced by the formation of volatile unsaturated compounds (*e.g.*, acetylene) when the melt is warmed with hydrochloric acid, so that more iodine is required than is equivalent to the hydrogen sulphide liberated. This error is eliminated by using, instead of commercial barium peroxide, hydrated barium peroxide containing 8 mols. of water of crystallisation prepared by dissolving barium hydroxide in water, filtering, precipitating with hydrogen peroxide, and drying the precipitate. W. T. K. Braunholtz.

Determination of moisture in solid fuels. D. J. W. Kreulen (Chem. Weekblad, 1926, 23, 476—477).—A Russian anthracite was found to contain 5·26% of combined water and 3% of volatile matter, calculated on the pure coal substance. In determinations of combined water made at temperatures over 105° a correction for oxidation is desirable; where exact values are required, the xylene method should be used. S. I. Levy.

Degree of swelling of caking coals, with particular reference to the geological and tectonic conditions of the coal seams. K. Patteisky (Brennstoff-Chem., 1926, 7, 315—316).—The degree of swelling of the bright coal, representing the bulk of the coal, and containing 18—30% of volatile matter, is about the same for coals from a variety of seams, and is a function of the chemical composition of the coal (*i.e.*, nature of the parent substance and tectonic conditions), but is independent of its geological age. The contrary conclusion arrived at by Dolch (cf. B., 1926, 729) is ascribed to the latter having considered an insufficiently wide variety of coals. W. T. K. Braunholtz.

[Degree of swelling of caking coals etc.] M. Dolch (Brennstoff-Chem., 1926, 7, 316).—A reply to Patteisky (preceding abstract). It is incorrect to neglect the influence of geological age on the degree of swelling of coal and to regard the latter as a function only of tectonic conditions and the still unknown chemical nature of the parent substances. Patteisky also goes too far in stating that the chemical properties of a coal, including its content of bituminous oil and degree of swelling, follow more or less necessarily from its content of volatile matter. W. T. K. Braunholtz.

Factors affecting carbonisation in continuous vertical retorts. T. F. E. Rhead (Gas J., 1926, 175, 843—859).—Experiments were carried out in a single Woodall–Duckham retort, using an improved governing device for controlling the gas pressure at the top of the retort. Imperfect governing of pressures within the retort causes alternate leakage of coal gas and furnace gas through the retort walls, thereby reducing the life of the plant and also promoting scurf formation. Two typical coals, one slightly swelling and the other strongly swelling, were examined. With slightly swelling coal, as normally crushed, the gas quality showed wider variations than were expected. This was attributed to irregularities in the movement of the charge within the retort, brought about by variations in the strength of coke causing irregular discharge. The coal entering the retort was uniform in quality, but varied in size on account of segregation within the bunkers. To avoid these irregularities in further tests, the coals were graded and supplied in small batches to the retort hopper. Comparing, under the same conditions of temperature, steaming, and throughput, coal graded between 3 in. and $1\frac{1}{4}$ in. with the same material crushed more finely, the larger coal gave a more porous charge which travelled uniformly and allowed freer circulation of the steam and gas, thus giving higher steaming and carbonising efficiencies, higher thermal yields of gas of uniform but slightly lower quality, higher tar and liquor yields, lower gas off-take temperatures, and steadier bottom pressures. The coke from large coal, however, was smaller and mechanically weaker than that made from crushed coal, and, whereas increased steaming of the charge from crushed coal did not materially affect the quality of the coke, steaming appeared to weaken the coke produced from larger coal and gave more breeze. Similar differences were exhibited between the same gradings of strongly swelling coal, but in both cases, compared with slightly swelling coal, the coke was larger and stronger. From trials of a number of methods of feeding coal into the retort it was found that by keeping the level of coal below the top of the retort the amount of rodding was considerably reduced. Experiments in which the heat distribution along the retort was varied showed that strongly swelling coals work well if "two stage" heating is adopted, *i.e.*, when the hottest part of the flue is midway down the retort, but not with high "top-heats." Less strongly swelling coals work equally well with either system, but with both classes of coal more efficient steaming and higher thermal yields are obtained with the maximum temperature near the middle of the retort. "Steam quenching" produced dry coke, but created a dust nuisance during discharging. Experiments on the production of water gas in vertical retorts filled with coke led to the suggestion that retorts requiring scurfing might be utilised with the double object of removing scurf and producing water gas. Attempts to crack vertical-retort tar in a stream of water gas by spraying the tar into the coke charge were unsatisfactory. S. Pexton.

Carbonisation plant at the Leopold Collieries, Edderitz. A. Sander (A. E. G. Mitt., 1926, 175—178; Chem. Zentr., 1926, II, 302).—A description is given of Geissen's lignite retort, working on the scavenging gas principle with internal heating. A closed vertical cast-iron drum rotates once in 3 minutes, and is enclosed in a stationary perforated gas-extracting drum. The material sinks downward in the space between the two drums, and is carbonised by the gases of combustion of gas burners, situated in the rotary drum and supplied with the carbonisation gases. The moisture in the latter is reduced from 50% to 5% by steam drying, and dust is removed before they are used for steam raising. The distillates are condensed, and tar, sulphurous water, benzine, and gases for heating separated. By arranging an upper and lower exit for the gases, rich gas and poor gas may be separately obtained. The space occupied by the retort for the same output is 1/10th that of a roller retort. H. Moore.

Influence of the ash constituents in the carbonisation and gasification of coal. C. B. MARSON (Gas J., 1926, **176**, 226—227).—A brief account of work already published (cf. B., 1925, 620). A. B. MANNING.

Carbonisation of wood. L. FAUQUE (Chim. et Ind., 1926, **16**, 544—551).—A description of carbonising and by-product recovery processes and plant.

Thomas gas calorimeter. Factors affecting its precision, flexibility, and reliability. R. A. RAGATZ and O. L. KOWALKE (Ind. Eng. Chem., 1926, **18**, 1087—1090).—The Thomas gas calorimeter, in which the heat of combustion of the gas is absorbed by a current of air, is practically independent of changes in the temperature of the tank, the atmospheric pressure, or the meter speed. These factors affect only the vapour pressure of the water in the meters, and this is compensated for by the temperature effect on the nickel wire used in the resistance thermometers. Comparative tests with a Junkers calorimeter showed a mean difference of 0·49% with various qualities of gas. The lag on variation in gas quality was found to be about 4 min. Variations in room temperature, atmospheric humidity, and in initial gas pressure from 2 to 8 in. water gauge, were found to have no appreciable effect. C. IRWIN.

Propagation of flame in mixtures of methane and air. IV. Effect of restrictions in the path of the flame. W. R. CHAPMAN and R. V. WHEELER (J.C.S., 1926, 2139—2147; cf. Mason and Wheeler, B., 1920, 775 A).—The velocity of a flame travelling in a methane-air mixture contained in a horizontal tube with a constriction decreases as the latter is approached, but increases again immediately before it is reached. Considerable acceleration occurs beyond the constriction. The same phenomena occur when the tube has a series of constrictions, provided they are spaced sufficiently, otherwise no appreciable retardation of velocity occurs between any two constrictions, though a large acceleration occurs after each is passed. The maximum flame-speed, 420 m./sec., was obtained when 12 constrictions are present irrespective of the length of the tube and the position of the first constriction; the flame under these conditions has a much greater actinic power, and a maximum pressure of 3·9 atm. is developed just beyond the last constriction. The effect of the constriction on the velocity of the flame is explained as the effect on the velocity of the current of gas which is shown experimentally to precede the travelling flame. S. K. TWEEDY.

Petroleum. III. Acid sludge from refining of Persian kerosene. I. Aromatic hydrocarbons. S. F. BIRCH and W. S. G. P. NORRIS (J.C.S., 1926, 2545—2554).—The dilution of the tar obtained by washing kerosene with fuming sulphuric acid (20% SO_3) gave an oil containing sulphur compounds and aromatic hydrocarbons. The presence of the following hydrocarbons was demonstrated by the preparation of suitable crystalline derivatives: toluene, *m*- and *p*-xylenes, mesitylene, ψ-cumene, hemimellithene, *p*-ethyltoluene, an ethylxylene, a diethylbenzene, naphthalene, β-methylnaphthalene, a dimethylnaphthalene, and some indication of a trimethylnaphthalene. A satisfactory method for the identification of aromatic hydrocarbons is to replace all the nuclear hydrogen atoms by bromine, with subsequent examination of the crystalline bromo-compounds. H. BURTON.

Catalytic formation of petroleum hydrocarbons from fats. J. MARCUSSON and W. BAUERSCHÄFER (Petroleum, 1926, **22**, 815—816; cf. B., 1926, 117).—The conversion of fats occurs at lower temperatures than those employed by Mailhe, the products obtained by whom were secondary. At low temperatures, avoiding distillation, heavy viscous oils are obtained as a primary product. Oil or fat was mixed with 20% of its weight of kieselguhr or sodium chloride and boiled for 20 hrs. under a reflux condenser, the temperature not exceeding 300°. The product, freed from catalyst and saponifiable matter, was fluorescent, had *d* 0·92 or 0·87, and iodine value 52 or 29 with linseed oil or tallow respectively as raw material. The acetyl values were 13 and 16, and the acetyl values of the hydrogenated products 32 and 61 respectively. H. MOORE.

Apparatus for examining oils, particularly motor lubricating oils for absorbed petrol ["crank-case dilution"], by distillation with steam. H. KIEMSTEDT (Brennstoff-Chem., 1926, **7**, 309—311).—In this compact modification of the usual apparatus for distilling oils with steam a cylindrical vessel containing the oil is held suspended in the neck of the steam generator by a conical plug. The oil vessel is thus steam jacketed, and the steam is introduced into the oil through a number of ∩-tubes, one limb of which is slightly longer than the other, and passes through the bottom of the oil vessel. This combined still is connected with a spiral condenser and a graduated receiver, from which the condensed water is automatically siphoned off, the whole apparatus being attached to a single retort stand. The steam generator carries a release cock, which is opened at the end of the distillation to prevent oil being drawn back into it. The apparatus may also be used for determining the water content of oils, in which case toluene or xylene is boiled in the generator instead of water. W. T. K. BRAUNHOLTZ.

"Saturation" of the petroleum lubricant hydrocarbons as shown by their reaction with bromine. C. F. MABERY (J. Amer. Chem. Soc., 1926, **48**, 2663—2664).—When treated with bromine in carbon tetrachloride, a sample of Illinois oil, b.p. 280—282°/30 mm., and one of Pennsylvania oil, b.p. 275—280°/30 mm., each yielded a *monobromo*-derivative, with evolution of 1 mol. of hydrogen bromide. These derivatives react readily with alcoholic potassium cyanide, the resulting nitriles being hydrolysed to acids when boiled with aqueous sodium hydroxide. It appears, therefore, that these oils do not contain unsaturated constituents. Russian and Texas oils behave similarly. F. G. WILLSON.

Counter-current jet condenser. B. MÜLLER (Petroleum, 1926, **30**, 1140—1142).—The condensers (Borrmann system) consist of a scrubbing-tower fitted with layers of irregularly packed material supported on perforated plates. The improvement consists in the perforations which are formed into jets projecting alternately upwards and downwards, thus separating

the flow of vapours and steam upwards from the flow of water downwards. W. N. Hoyte.

Determination of sulphur in liquid fuels. R. de Fazi (Annali Chim. Appl., 1926, 16, 405—406).—The Eschka method, which is one of the methods approved by the Italian Government Commission for the determination of sulphur in liquid fuels, gives results considerably lower than the concordant figures obtained by the Mahler bomb method and Gasparini's method (B., 1908, 340), and its abandonment is recommended. T. H. Pope.

Apparatus for determination of water in oils etc. N. Schapiro (Chem.-Ztg., 1926, 50, 579).—The apparatus embodies the ordinary distilling flask and condenser, but is made in one piece, and is provided with a tubulus for filling. S. I. Levy.

Dielectric constant of petrols and paraffins. H. Pécheux (Compt. rend., 1926, 183, 530—532).—The dielectric constants ϵ and their temperature coefficients α, and the m.p. and b.p. of six petroleum oils and three paraffins have been determined. For liquid petrols and heavy paraffins respectively, α (which is negative) increases and decreases in absolute value with ϵ. In both cases ϵ increases with the density. For paraffins the value of α is one-half to one-third that for liquid petrols. J. Grant.

Volumetric determination of acetylene. R. Strebinger and A. Wojs (Z. anal. Chem., 1926, 69, 20—29).—A measured volume of the gas is passed at the rate of 1 c.c. in 2 min. through two U-tubes in succession, each maintained at 190—200°, and containing glass beads moistened with a solution of 2 g. of potassium iodate in 20—23 c.c. of concentrated sulphuric acid. When all the gas has passed through the tubes the apparatus is swept out by means of a slow current of air. The U-tubes are rinsed out with water, the solution is diluted to 400—600 c.c. and boiled until colourless, and the excess of iodate titrated with $0 \cdot 1N$-thiosulphate after cooling. The reaction is $C_2H_2 + 2KIO_3 + H_2SO_4 = 2CO_2 + 2H_2O + I_2 + K_2SO_4$. Traces of hydrogen and carbon monoxide are sometimes present in commercial acetylene, but the amount of iodate consumed by them is very small. A. R. Powell.

Sulphur content of mixed gas in the Siemens-Martin furnace. W. Hülsbruch (Gas- u. Wasserfach, 1926, 41, 886—888).—See B., 1926, 859.

Patents.

Washing coal and other minerals. A. France (E.P. 251,246, 24.3.26. Conv., 23.4.25).—The material to be washed is carried along a bed by a current of liquid and jets of water play up through it from the bed and separate it into layers according to its specific gravity. Three layers are discriminated—clean coal, intermediate products, and waste products. They are separated in turn, the intermediate products being returned for re-washing. The process is particularly suitable for materials containing but little of the waste material of high specific gravity. In this case a suitably-sized foreign material of much the same specific gravity as the waste product is added, and the waste product is then assimilated into the layer which the added product forms. R. A. A. Taylor.

Coal concentration. W. Truran, Assr. to Minerals Separation North American Corp. (U.S.P. 1,595,745, 10.8.26. Appl., 24.5.22).—The mixture of coal and water is first aerated alone, and the froth separated, and then an oil is added and the mass aerated again, the froth being again separated. The two froths are mixed and aerated in the presence of starch, by which means a very clean froth is obtained. E. S. Kreis.

Differential coal flotation. J. V. Quigley, Assr. to Minerals Separation North American Corp. (U.S.P. 1,595,731, 10.8.26. Appl., 24.5.22).—In the process described in U.S.P. 1,595,745 (preceding abstract) the froth obtained by aeration without the addition of an agent is separated and the coal contained in it recovered separately, as it contains bright coal, with a much lower percentage of sulphur than the original. The coal in the froth obtained by aeration with the assistance of an oil is a dull coal, and this is also recovered separately. E. S. Kreis.

Washing coal and ores. L. Hoyois (E.P. 258,753, 28.11.25).—Coal carried by a stream of water is caused to pass over a weir, and then enters a trough having openings of adjustable size suitably distributed along its length. The openings allow successively lighter fractions to be collected, and the raw material can be graded into a number of products having densities defined within narrow limits. S. Pexton.

Treatment of peat. F. M. Perkin (E.P. 258,419, 8.9.25).—Peat is elevated to a macerator consisting of pairs of counter-running toothed rollers, which are hollow and can be heated if desired. The disintegrated peat is forced through a screw conveyer, working in a perforated cylinder within an outer chamber which collects the extruded water into an autoclave. When this is suitably charged it is closed, superheated steam is admitted, and the mass is agitated. The pressure in the autoclave rises to a maximum, and the contents are then suddenly discharged through a valve communicating with a receiving chamber, where a large proportion of original water in the peat passes away as steam. The bottom of the receiving chamber is provided with a screw conveyer, which transfers the peat under pressure to a centrifuge or filter press, where it is briquetted. Using raw peat containing 80% of water, the moisture content of the briquetted product is 18—20%. S. Pexton.

Manufacture of active carbon. J. Nagtegaal (E.P. 257,766, 14.10.25).—The carbonaceous material is distilled in retorts situated in a furnace; the distillation gases are led back to the furnace for combustion, and the products directly into the same or another retort through apertures in the retort wall, this being effected by maintaining in the retort a pressure below that prevailing in the furnace. A. C. Monkhouse.

Drying and low-temperature distillation of fuel by internal heating. Metallbank u. Metallurgische Ges. A.-G. (E.P. 242,618, 22.10.25. Conv., 4.11.24).—The plant comprises one or more distillation chambers, together with furnaces for providing heat which is

conveyed by circulating gases to the distillation chambers. Interposed between the furnaces and the distillation chambers are flues wherein the circulating gases are heated by admixture with the products of combustion from the furnaces. In this way heat is conserved, and local overheating of the distillation chambers is avoided. The temperature of distillation can be readily controlled and varied according to the proportion of combustion products in the circulating gases. S. PEXTON.

Retorts. KOHLENVEREDLUNG GES. M.B.H. (E.P. 245,773, 6.1.26. Conv., 8.1.25).—The retort consists of the annular space between two concentric cylinders. The inner is corrugated and designed to revolve, and the outer is provided with guide rings, inclined inwards and downwards, which direct the material to be carbonised against the wall of the inner cylinder as it revolves. The products of distillation pass off through slots under the guide rings into a second annular space between the outer cylinder and the outside wall of the plant. As the temperature of the charge rises in its path down through the retort, a partial fractionation of the distillation products may be effected by partition walls at varying heights in the outer annular space. The retort is heated by a flue carrying heating gas to the closed top of the inner corrugated cylinder, and the gas then passes down between the flue and the inner cylinder. R. A. A. TAYLOR.

Revoluble kilns for distilling bituminous materials. E. ROSER (E.P. 257,479, 29.12.25).—The retort is of the revolving drum type, in which the annular space is heated both internally and externally by heating gases either in parallel or in series. The annular drum is divided into partitions, so that as it revolves the gas is drawn off, either from each section into pipes passing along the inner chamber, or by one discharge pipe passing through the outer chamber. The operation is continuous, the coked material being withdrawn down a shoot as the drum rotates. A. C. MONKHOUSE.

Gas producer or generator. F. W. STEERE (E.P. 258,348, 17.6.25).—A gas producer is provided with one or more hollow pokers, which can be mechanically thrust upwards into the fuel bed from an air-tight ash-pit beneath the grate. The grate is composed of fixed and movable bars, the latter being raised and lowered by lugs on the movable pokers, to allow the clinker broken by poking to fall into the ash-pit. Another design of grate included comprises a series of toothed rotatable bars, geared externally, and operated by a lever system coupled to the movable poker so that, simultaneously with poking, the grate bars rotate and extract the crushed clinker. The position of the pokers, which are cooled by the air and steam which pass through them, can be so adjusted as to convey steam and air to any desired part of the fuel bed. S. PEXTON.

Conversion of heavy hydrocarbon oils into light hydrocarbon oils. F. LAMPLOUGH, and RATOCZYN EXTENDED OIL FIELDS, LTD. (E.P. 258,656, 23.6.25).—Heavy hydrocarbon oils mixed with 0·4% of an aromatic hydrocarbon such as naphthalene are heated at about 600° under a pressure higher than the vapour pressure of the oils at that temperature. The apparatus for treatment consists of two steel pipes interconnected and coiled about a vertical axis, the inner pipe being smaller in diameter than the outer one. The coils are directly heated, or preferably heated in a bath of molten metal. The oil is delivered to the inner coil by means of a pressure pump, and is either circulated slowly or treated statically. To the outlet of the outer coil are connected a cooler and a spring-operated release valve. The products of treatment are cooled under pressure to such a temperature that the heavier fractions remain liquid whilst the light hydrocarbons are discharged from the release valve as vapours and are separately condensed. S. PEXTON.

Continuous production of low-boiling-point hydrocarbons from hydrocarbons having a high boiling point. H. WOLF (E.P. 252,308, 6.7.25. Conv., 22.5.25).—Hydrocarbons are cracked under pressure and the products passed into a suitable solvent or wash oil, when the free carbon and highly carbonaceous substances, which would otherwise choke the apparatus, are held in suspension or solution. The mixture is distilled and the residue of heavy oils returned to the cracking process, or the lighter portions of the cracked products may be distilled off below the boiling point of the wash oil, which may be used again until fully charged with the detrimental carbonaceous substances, when it is run off. R. A. A. TAYLOR.

Lubricating oil. A. SCHELLER (Can. P. 256,104, 9.5.22).—High-boiling oils are distilled in the presence of small quantities of alkaline oxides or hydroxides, without further drying or chemical or physical refining. H. MOORE.

Making low-boiling hydrocarbons from petroleum. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,597,796, 31.8.26. Appl., 13.11.19).—The mixture of partially oxidised hydrocarbons (cf. E.P. 209,128; B., 1924, 210), produced by passing a vaporised hydrocarbon mixture admixed with the necessary quantity of air over a catalyst, is condensed, and the lighter fractions are separated. The remainder is vaporised, mixed with steam, and passed over a catalyst, *e.g.*, copper, at 450°. The metal is oxidised by the steam, with the liberation of hydrogen, which reacts with the hydrocarbon derivatives to give saturated hydrocarbons of lower molecular weight than the original constituents of the petroleum. According to the reactions shown, carbon monoxide is liberated in most of these reactions and is probably responsible for the reduction of the catalyst, since this always contains some free metal to combine with the oxygen of the steam and continue the reaction. E. S. KREIS.

Oxidising heavy hydrocarbons. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,597,797, 31.8.26. Appl., 8.12.21).—To oxidise a heavy mineral hydrocarbon (cf. E.P. 209,128; B., 1924, 210) it is mixed with a light petroleum and the mixture vaporised with the aid of steam. In this way, wax distillate, pressure tar, and heavy lubricating oils have been vaporised and passed through the catalyst screens without depositing on them and clogging them. It is found that in the vapour phase, the hydrocarbons of high mol. wt. are oxidised preferentially. The best results are obtained when only a portion of the necessary oxygen is added in the

vaporising chamber, the remainder being added after the mixture has passed through the first screen.

E. S. Kreis.

Treating liquid hydrocarbons. J. H. James, Assr. to C. P. Byrnes (U.S.P. 1,597,798, 31.8.26. Appl., 21.7.22).—The partially oxidised hydrocarbons (cf. E.P. 209,128; B., 1924, 210) are condensed, and the lighter portion, boiling up to 200°, is separated and used as fuel for internal-combustion engines, for which, after it has been purified from acids by distilling from 5% of dry sodium hydrate, it possesses the advantages of being easily combustible and anti-knock. The heavier portion is mixed with fresh untreated petroleum and run through the cycle again, the lighter portion again being separated.

E. S. Kreis.

Treating petroleum emulsions. M. de Groote and W. C. Adams, Assrs. to W. S. Barnickel & Co. (U.S.P. [A] 1,595,455 and [B] 1,595,456, 10.8.26. Appl., 6.4.25).—(A) Petroleum is separated from emulsions by the addition of a compound formed from a "modified" carbohydrate and a dissimilar substance, such as a fatty acid, a resin, or naphthenic acid. In an example, 1 pt. of the carbohydrate is treated with 15 pts. of sulphuric acid at a temperature not exceeding 60°, and 50 pts. of this mixture are heated with 100 pts. of oleic acid at a temperature not exceeding 80°; the resulting mass after neutralisation is treated with a solubilising agent which will give a dispersion in either oil or water. (B) 1 pt. of a protein is treated with 15 pts. of sulphuric acid, and then, after heating with oleic acid, used in the same way.

E. S. Kreis.

Treating petroleum emulsions. M. de Groote and E. H. Keiser, Assrs. to W. S. Barnickel & Co. (U.S.P. 1,595,457, 10.8.26. Appl., 6.4.25).—The emulsion is broken down with the aid of the insoluble salt of a modified organic soap-forming material. For instance, rosin dissolved in oleic acid is sulphonated, and the alkaline-earth or other salts of the product are used to treat the emulsion.

E. S. Kreis.

Recovery of lime products from the manufacture of acetylene gas (E.P. 258,660).—See VII.

III.—TAR AND TAR PRODUCTS.

Patent.

Hydrogenating tar oils. W. Demann (E.P. 236,230, 26.6.25. Conv., 30.6.24).—Dehydrated tar oil, suitably desulphurised, is hydrogenated at ordinary pressure by heating in shallow pans and bubbling in, in the presence of a suitable catalyst, coke-oven gas, which must necessarily be dry.

R. A. A. Taylor.

IV.—DYESTUFFS AND INTERMEDIATES.

Patents.

Manufacture of wool dyestuffs of the anthraquinone series. Farbenfabr. vorm. F. Bayer & Co. (E.P. 240,492, 28.9.25. Conv., 27.9.24).—Unsubstituted 1 : 1′-dianthraquinonylamine is sulphonated to a mono- and a di-sulphonic acid. *E.g.*, a solution of 10 pts. of 1 : 1′-dianthraquinonylamine in 100 pts. of 15% oleum is heated at 95—100° until a sample is completely soluble in hot water. After cooling and pouring into 600 pts. of water, the dye is salted out. It dyes wool yellowish-brown, unaltered by chrome mordants and fast to light, fulling, and carbonising. It is believed to be 1 : 1′-*dianthraquinonylamine*-4 : 4′-*disulphonic acid*, since on treatment with bromine in aqueous solution it yields 4 : 4′-*dibromo*-1 : 1′-*dianthraquinonylamine*. By sulphonation at 30° with 25% oleum a new *monosulphonic acid* is obtained, which dyes wool brownish-red. (Cf. E.P. 201,575, B., 1924, 861.)

A. Davidson.

Preparation of [stable water-soluble derivatives of vat] dyes. Scottish Dyes, Ltd., J. Thomas, J. E. G. Harris, and B. Wylam (E.P. 258,626, 17.3.25).—Of the known processes for the preparation of derivatives of leuco-compounds of vat dyes (cf. E.P. 23,396 of 1899; 186,057, 202,630, 202,632, 245,587, 247,787, 248,802; B., 1900, 1101; 1924, 252, 375; 1926, 235, 403), those in which the leuco-compound is used as the starting material are improved in efficiency by the addition of a metal to the reaction mixture containing the leuco-compound, a suitable organic base and one of the following reagents, viz., chlorosulphonic acid and its salts, alkylsulphuric halides, oleum and its salts, sulphuric anhydride, alkylphosphoric halides, or phosphorus oxychloride. Suitable metals for this purpose are zinc, tin, magnesium and copper bronzes, or copper bronze mixed with 75% of zinc. *E.g.*, 5 pts. of dimethoxydibenzanthrone are reduced at 50° with 9 pts. of sodium hyposulphite and 18 pts. of sodium hydroxide in 800 pts. of water. The vat is filtered and, under hydrogen, is acidified, the leuco-compound filtered off, washed, and dried at 60° under a low pressure of hydrogen. 10 pts. of the dry leuco-compound are mixed with 10 pts. of zinc dust and stirred into a mixture made by slow addition of 34 pts. of methylsulphuric chloride to 75 pts. of well-cooled pyridine, in a vessel which may be open to the air. The melt is warmed to 60°, kept at this temperature for 30 min., and then stirred into 750 pts. of cold water. The precipitate is filtered off, washed, and extracted with 2—4% sodium carbonate solution at 40—50°. By adding salt to the filtered extract, a stable sulphuric acid derivative of dimethoxydibenzanthrone is precipitated. Other examples relate to the formation of derivatives of the leuco-compounds of indigo, thioindigo, indanthrone, and flavanthrone. The stable water-soluble products may be used for dyeing or printing textile fibres.

A. Davidson.

Manufacture of chloro- and chloronitro-derivatives of *m*-hydroxybenzaldehyde. Brit. Dyestuffs Corp., Ltd., and H. H. Hodgson (E.P. 258,060, 13.8.25).—*m*-Hydroxybenzaldehyde is chlorinated in cold glacial acetic acid until enough chlorine for a monochloro-derivative has been absorbed. Part of the 2-*chloro*-3-*hydroxybenzaldehyde*, m.p. 139·5°, which is formed crystallises out and the remainder is separated from accompanying 6-chloro-3-hydroxybenzaldehyde by fractional neutralisation of the evaporated residue with aqueous sodium carbonate. Further chlorination of the 2-chloro-compound yields 2 : 6-dichloro-3-hydroxybenzaldehyde, m.p. 140°. Nitration of the 2-chloro-compound in 50% acetic acid at 45—50° produces a mixture of 2-*chloro*-4-*nitro*-3-*hydroxybenzaldehyde*, m.p. 166°, and 2-*chloro*-6-*nitro*-3-*hydroxybenzaldehyde*, m.p. 154°, the

two being separated by steam distillation, when the volatile 4-nitro-compound distils. Nitration of the 2 : 6-dichloro-compound in glacial acetic acid at 60° yields 2 : 6-*dichloro-4-nitro-3-hydroxybenzaldehyde*, m.p. 80°.
A. DAVIDSON.

Preparing dyestuffs. BRIT. ALIZARINE CO., LTD., and C. M. BARNARD (E.P. 258,960, 4.7.25).—Amino-anthraquinones and -benzanthrones are condensed with citric acid. For example, 20 g. of 1 : 4-diaminoanthraquinone are boiled with 80 g. of crystallised citric acid under reflux for 1 hr., without a diluent. The condenser is then removed and heating continued at 150° until a sample is completely soluble in sodium carbonate solution. The melt is then poured into 500 c.c. of hot water, boiled, cooled, and filtered. The solid is dissolved in sodium carbonate solution or aqueous ammonia and the dye salted out or the solution evaporated to crystallisation. The product dyes acetyl silk a magenta colour.
A. DAVIDSON.

Sulphonation of aromatic amines. J. W. LEITCH & CO., LTD., and A. E. EVEREST (E.P. 257,979, 2.6.25).—Aromatic amines such as aniline, *o*- and *p*-toluidines, *o*- and *p*-chloroanilines, *o*- and *p*-anisidines, *p*-phenetidine α- and β-naphthylamines, *m*-xylidine, and mixed xylidines are sulphonated by treating with sulphuric acid their condensation products with aromatic sulphochlorides. For example, 100 lb. of *p*-toluenesulpho-*p*-toluidide are stirred into 314 lb. of 95·5% sulphuric acid and the mixture warmed to 73°, when a reaction begins which, without further external heating, raises the temperature to 87°. After cooling to 50°, the mass is stirred into 1200 lb. of water and allowed to stand, when 4-aminotoluene-3-sulphonic acid crystallises. If 21·6% oleum is used, the reaction may be started at 22°. The process is, however, not effective with benzidine, tolidine, dianisidine, aminoazobenzene, aminoazotoluene, *m*-nitro-*p*-toluidine, *m*- and *p*-nitroanilines, 4-chloro-3-nitroaniline, 4-chloro-3 : 5-dinitroaniline, or 3 : 5-dinitro-*p*-toluidine.
A. DAVIDSON.

Quantitatively halogenising perylene. A. PONGRATZ and A. ZINKE (E.P. 244,739, 28.11.25. Conv., 19.12.24).—Perylene in solution in nitrobenzene or the like is acted on by nascent halogen. For example, to 1 part of perylene dissolved in 20 parts of nitrobenzene at 90° is added, during 1½ hrs., from one dropping funnel a mixture of 2 parts of concentrated hydrochloric acid and 18 parts of glacial acetic acid, and from another a mixture of 0·9 part of 30% hydrogen peroxide and 8 parts of glacial acetic acid. On cooling, 3 : 9-*dichloroperylene*, brownish-yellow, m.p. 280°, separates. The preparation of 3 : 4 : 9 : 10-*tetrachloroperylene*, reddish-orange, m.p. 350°, and of a *hexachloroperylene*, yellow, m.p. 356—357°, by analogous methods is also described.
A. DAVIDSON.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Effect of ultra-violet light on wool. L. MEUNIER and G. REY (Compt. rend., 1926, **183**, 596—598).—Wool becomes more acid when exposed to ultra-violet light, and develops a yellow colour with quinone in place of the usual violet colour. Bleached wool gives the same yellow coloration. Irradiated and sulphur-bleached wool also fails to give the usual colours when treated with ninhydrin or allantoin owing to the presence of sulphur dioxide. The increase in labile sulphur content on irradiation is also shown by reduction of nitro-compounds to amines. Tips of wool which have been exposed to sunlight always behave differently on irradiation from wool situated nearer the skin.
L. F. HEWITT.

Comparison of "wetting-out" agents [for textile processes]. W. KIND and J. AUERBACH (Textilber., 1926, **7**, 775—780).—Further investigations are recorded (cf. Auerbach, B., 1926, 705) in which the wetting-out powers of many proprietary substances are compared by determining the rates of sinking of a square of dry woollen fabric in their aqueous solutions (*ibid.*), and also by means of their drop-numbers (measured by means of Traube's stalagmometer).
A. J. HALL.

Determination of deliquescent substances in sized cotton materials. S. M. NEALE (J. Text. Inst., 1926, **17**, T 511—516).—Methods are described for the determination of chlorides (Volhard), zinc, magnesium, and glycerol in sized goods, which yield trustworthy results using 5—10 g. of yarn or cloth or 1 g. of size. For the separation of magnesium and zinc, the former is precipitated as magnesium ammonium phosphate from an ammoniacal solution and the filtrate is boiled to expel ammonia and rendered just acid to bromocresol-purple with acetic acid, when zinc ammonium phosphate separates. These precipitates are converted into ammonium phosphomolybdate, which is collected and decomposed in an excess of *N*-sodium hydroxide, the unused alkali being titrated. Glycerol is determined by extraction and oxidation to carbon dioxide in a gas burette. Full details are given.
J. C. WITHERS.

Decomposition of cellulose under pressure, with and without a liquid medium and an atmosphere of hydrogen. H. I. WATERMAN and J. N. J. PERQUIN (Rec. trav. chim., 1926, **45**, 638—653; cf. A., 1913, ii, 579; J.S.C.I., 1925, 508 T).—Cellulose, in the form of cotton wool pellets, when heated under pressure at temperatures varying from 225° to 450° both in the presence and absence of hydrogen, was rapidly decomposed. At 225°, after 2 hrs.' heating, 85% of the cellulose was recovered as a black residue resembling coal (C 48·9%, H 6·1%). At 300°, after 1 hr. heating, only 54% of solid residue (C 71·0%, H 5·2%) was obtained, about half the cellulose being converted into carbon dioxide and water, together with a small amount of methane. At 450° a much more profound decomposition took place, the chief products being carbon dioxide, carbon monoxide, water, and methane and homologues, with 37% of solid residue (C 85·7, H 5·0%). The nature and quantity of the reaction products were not greatly affected by the presence of hydrogen, although the amount of hydrocarbons formed was increased. There was a considerable evolution of heat during decomposition. The heat of combustion of the solid residue obtained at 450° was 7832 g.-cal./g., against 4158 g.-cal./g. for the cellulose used. On heating the cellulose in the presence of a liquid medium (Borneo petroleum) an increase in the yield of methane and homologues and a decrease in the amount of solid residue resulted. Cellulose was also heated *in vacuo*

at 300°, and 38% of the material was recovered as a viscous liquid considered to be an intermediate product between cellulose and the solid residue obtained in the experiments under pressure. W. J. POWELL.

Decomposition of cellulose in the soil. S. WINOGRADSKY (Compt. rend., 1926, **183**, 691—694).—A description of the appearance of *Spirochæta cytophaga* (Hutchinson and Clayton, J. Agric. Sci., 1919, 9, 143) in the various stages of its action on fibres of pure cellulose. The cellulose is transformed first into a translucent colloidal substance, believed to be an "oxycellulose." G. M. BENNETT.

Non-volatile constituents of the cotton plant. POWER and CHESNUT.—See XVI.

Deliquescent properties of magnesium chloride, calcium chloride, and glycerol. SWAN.—See VII.

Chemical characteristics of some trade wastes. BUSWELL, GREENFIELD, and SHIVE.—See XXIII.

Disposal of some organic trade wastes. BARTOW.—See XXIII.

PATENTS.

Washing or cleaning wool. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (E.P. 256,635, 2.3.25. Conv., 13.5.24).—Wool is washed by treatment with a concentrated suint liquor (*d* 1·029) in a machine which comprises a succession of washing bowls each followed by squeezing rollers, the wool passing continuously through at least four bowls. The suint liquors are maintained clean by periodically flushing out the mud which accumulates under the false bottoms of the bowls, and their concentration is maintained constant by the introduction of suint or soluble salts recovered from the wool after emerging from the washing process (cf. E.P. 240,482, 243,499; B., 1925, 985). A. J. HALL.

Manufacture of cellulose acetate. H. J. MALLABAR (E.P. 258,020, 17.6.25).—Cellulose acetate which is soluble in acetone, free from sulphuric acid residues, and does not char when heated, is obtained by acetylating cellulose in the absence of sulphuric acid, but after a pre-treatment with a mixture of acetic acid and sulphuric acid, the sulphuric acid being subsequently neutralised before acetylation proper is effected. Zinc chloride is used as a catalyst during the acetylation. For example, 100 pts. of cellulose are treated for 24 hrs. with a cold mixture of 400 pts. of acetic acid and 2—5 pts. of sulphuric acid, and then 200 pts. of acetic acid, containing sufficient sodium acetate to convert the sulphuric acid into sodium sulphate, are added; subsequently 200 pts. of acetic acid, 15—25 pts. of zinc chloride, and 250—400 pts. of acetic anhydride are added, and the mixture is maintained at 30—40° until acetylation is complete. The resulting cellulose acetate may be precipitated directly from the product and afterwards converted into an acetone-soluble form by dissolving it in acetic acid, and maintaining the solution at 100°. A. J. HALL.

Production of artificial yarns or threads. SOC. POUR LA FABR. DE LA SOIE RHODIASETA (E.P. 251,580, 18.6.25. Conv., 28.4.25).—The shading of artificial silk with fugitive dyes in order to distinguish fibres of different nature and characteristics is effected, in the case of threads produced by the dry-spinning process, before, during, or after the association of the elementary filaments into a thread, or during the reeling operation. When the supply of dyes is limited and a large number of fibres of different character require differentiation, systematic irregularities may be introduced; for example, some of the elementary filaments of the thread may be dyed, while the other filaments remain undyed or are coloured with another dye. The shading device may be placed inside or outside the spinning chamber, and may consist of a roller, which is continuously impregnated with a solution, *e.g.*, 1%, of acid carmine blue V, azonaphthol red 6B, brilliant milling green NB, or the like. Mineral colours, such as ultramarine, may also be used, and are conveniently applied by spraying. D. J. NORMAN.

Preparing artificial silk yarn. S. TODA (E.P. 243,009, 13.11.25. Conv., 14.11.24).—Untwisted viscose silk yarn is made by passing freshly-coagulated and washed viscose filaments in a bundle through a sizing bath, optionally containing softening agents. A suitable sizing bath is obtained by boiling 1 part of *Gloiopeltis* (a Japanese sea weed known as "funori"), 1 part of dextrin, 0·16 pt. of gelatin, and 0·5 pt. of wax in 100 pts. of water. The sized yarn is then reeled in the usual way. The protective coating of size prevents fluffing during weaving, and, when subsequently removed in the desulphurising and bleaching baths, leaves a fabric of softer feel than is obtained with twisted viscose silk yarn. D. J. NORMAN.

Reducing the viscosity characteristics of nitrocellulose. W. R. WEBB, Assr. to EASTMAN KODAK CO. (U.S.P. 1,598,949, 7.9.26. Appl., 11.2.25).—Nitrocellulosic material is treated with an aqueous liquor containing an acid and a wetting-out agent (an organic liquid) until the material has the desired reduced viscosity characteristics. A. J. HALL.

Method of impregnating cellulose fibres. W. C. CARTER (U.S.P. 1,598,640, 7.9.26. Appl., 22.7.24).—The depth of superficial formation of cellulose xanthate within paper stock is controlled by limiting the swelling which accompanies the reaction. A. J. HALL.

Manufacture of a derivative of cellulose. S. A. OGDEN (E.P. 246,476, 20.1.26. Conv., 20.1.25).—Cellulose is treated with sulphuric acid of 50—75% strength, at a temperature not less than 45°, nor exceeding 70°, the duration of the treatment depending on the strength of the acid, the temperature, and, under certain conditions, the pressure. The reaction product is obtained in paste form, and when washed free from acid, is an irreversible colloid which dries into a horny mass without the formation of acid salts on its surface. In its colloidal form this product is suitable either as a parent material for the manufacture of other cellulose derivatives, or as a binding agent for clay, paper, asbestos, rubber, etc. *Example:* Cellulosic material, preferably cotton, is agitated for about 20 min. in a bath of 64·8% sulphuric acid by weight at 70°. The resulting product is then washed until sufficiently free from acid to dry without

the appearance of acid salts on its surface. When the primary material contains animal fibres, which it is desired to recover, the mixed fabric is treated for not more than 5 min. in a bath of 73—84% sulphuric acid (by weight), at 60—70°; separation of the animal fibre is then effected in the first washing operation.

D. J. NORMAN.

Nozzles for use in the production of artificial filaments, threads, and the like. COURTAULDS, LTD., and J. E. CRIGGAL (E.P. 258,365, 23.6.25).—The nozzles are made by heavily compressing wire gauze until the perforations are reduced to the desired size.

D. J. NORMAN.

Treatment of waste paper and the like in the preparation of paper pulp. J. F. L. MOELLER (E.P. 258,630, 24.3.25).—Apparatus is described for de-inking waste paper, using preferably cold water as a cleansing agent, progressive mechanical disintegration followed at each stage by violent agitation with water being relied upon to loosen the inky materials. The process is continuous, the pulp being reduced in fineness until it is clean and sufficiently milled to run on a paper machine. D. J. NORMAN.

Production of electrical insulating [fibrous] material (E.P. 236,224).—See XI.

Treatment of soda solutions contaminated with cellulose (E.P. 249,091).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Colour problems in the woollen and worsted industries. S. G. BARKER and H. R. HIRST (J. Text. Inst., 1926, **17**, T 483—510).—A survey is given of present knowledge concerning fading problems. New information includes the results of exposing patterns dyed with Victoria Blue to sunlight during the separate months January—June, and under five different cloud conditions. Full sunlight caused a fairly constant relative fading, and dull light had about one-tenth of the effect of full sun. In addition, comparative tests on dyed worsteds exposed to flame arcs, sunlight in Leeds, and sunlight in a country place near Leeds are recorded for 86 dyes. J. C. WITHERS.

Standardisation of methods of testing the fastness of dyes. A. CRUMMETT (J. Soc. Dyers and Col., 1926, **42**, 301—304).—Some notes on the testing of dyed natural silk as regards its fastness to light, water, washing (scouring), degumming, and perspiration. A. J. HALL.

Absorption of water by dried films of boiled starch. SWAN.—See XVII.

PATENTS.

Bleaching textile materials by the "pack" system with circulating liquor. F. KIESER (E.P. 258,448, 24.11.25).—A bleaching liquor is uniformly distributed in drops or in thin trickling streams from a sprinkler which is continually moved above the surface of the textile material contained in a suitable vat, the amount of liquor thus distributed being equal to that withdrawn from the bottom of the vat, and such that the textile material is maintained saturated with the bleaching liquor. The necessity for pressure or suction is thus avoided, and easy passages for the bleaching liquor through the textile material are not formed.

A. J. HALL.

Dyeing [cellulose] acetate silk. BRIT. DYESTUFFS CORP., LTD., R. S. HORSFALL, L. G. LAWRIE, J. A. R. HENDERSON and J. HILL (E.P. 257,654, 29.5.25).—Water-soluble dyes which may be used for dyeing cellulose acetate silk without the use of a solubilising agent (cf. E.P. 219,349; B., 1924, 906) are obtained from many acid dyes by converting their sulphonic acid groups into sulphonamide groups (*e.g.*, by successive treatment with phosphorus pentachloride and ammonia), or, alternatively, by synthesis of dyes of the acid type from components containing sulphonamide groups. Reddish-orange and orange dyes are obtained by coupling diazotised aniline with 1-naphthol-3 : 8-disulphonamide or 1 : 8-naphthasultam respectively.

A. J. HALL.

Dyeing cellulose acetate products in [oxidation] black shades. SILVER SPRINGS BLEACHING AND DYEING CO., LTD., and A. J. HALL (E.P. 258,699, 10.7.25).—Cellulose acetate silk is dyed in very fast black shades by application of 2 : 4-diaminodiphenylamine from aqueous solutions or suspensions and subsequent immersion in hot or boiling aqueous solutions of oxidising agents (cf. E.P. 246,879; B., 1926, 317), or by impregnation with a paste containing an oxidising agent, a catalyst, an acid, and 2 : 4-diaminodiphenylamine followed by exposure to a moist warm atmosphere for development of the black shade. Mixtures containing 2 : 4-diaminodiphenylamine and one or more aromatic amines such as aniline, *o*-toluidine, *p*-phenylenediamine, and *p*-aminodiphenylamine may be used. A. J. HALL.

Methods and apparatus for dyeing, colouring, or coating materials [fabrics]. TWO-TONE CORP., Assees. of P. MIJER (E.P. 236,194, 9.6.25. Conv., 30.6.24).—Textile fabrics are passed transversely through a cloud produced by atomising a solution of a dye or coating composition, and are thereby coloured or coated on one side. The fabric may be calendered or heated before passage through the cloud and afterwards steamed and dried. A. J. HALL.

[Re-lustreing] treatment of threads, fabrics, or other materials containing [cellulose acetate] artificial filaments. BRIT. CELANESE, LTD., C. W. PALMER, and S. M. FULTON (E.P. 259,266, 26.5.25).—Cellulose acetate silk threads which have lost their natural high lustre by immersion in boiling aqueous liquors are re-lustred to any desired degree by immersion in boiling, or nearly boiling, aqueous solutions of neutral salts (cf. E.P. 246,879; B., 1926, 317) or sugars. Suitable salts include the sulphates and chlorides of ammonia, sodium, potassium, and aluminium. For example, de-lustred cellulose acetate silk is re-lustred by immersion for 10—40 min. in a boiling aqueous solution containing 2—3% of ammonium sulphate or 5—10% of cane sugar. Alternatively, the de-lustred silk is subjected to the action of a swelling agent, *e.g.*, ammonium thiocyanate (cf. E.P. 158,340; B., 1921, 213 A), before treatment with the re-lustreing solution. A. J. HALL.

Treatment [mercerisation] of cotton fabrics containing [cellulose acetate] artificial silk. CALICO

PRINTERS' ASSOC., LTD., and L. A. LANTZ (E.P. 259,394, 29.10.25).—In the mercerisation at the usual temperatures of cotton fabric containing cellulose acetate silk, protective substances such as salts of alkali metals, aluminium, zinc, or inorganic or organic acids, polyhydric alcohols, carbohydrates, or phenols are added to the mercerising liquor for the purpose of preventing deterioration (by hydrolysis) of the cellulose acetate silk. Suitable protective substances include sodium chloride, chlorate, sulphate, and glycollate, zinc oxide, chloride, and sulphate, sucrose, glycerol, phenol, and resorcinol. For example, a satisfactory mercerising liquor consists of a solution containing 10—15% of sodium chloride and 15—20% of sodium hydroxide (cf. E.P. 210,484; B., 1924, 290). A. J. HALL.

Treatment [with synthetic resins] of cotton, silk, or artificial silk or goods made therefrom. BRIT. CYANIDES CO., LTD., E. C. ROSSITER, and W. C. DAVIS (E.P. 258,357, 19.6.25).—Synthetic resins, particularly those made from formaldehyde and thiourea, and which in their first stage of condensation are water-soluble, are used, together with fillers such as starch, gums, china clay, etc., for stiffening textile materials. Thus, cotton goods are impregnated with a solution containing fillers, a water-soluble condensation product of thiourea and formaldehyde, and a condensing agent such as an acid or salt thereof (*e.g.*, potassium tetroxalate) or the hydrochloride of an organic base, and then subjected to a high temperature (not exceeding 120—140°) whereby the condensation product is rendered water-insoluble. A. J. HALL.

Dyeing of cellulose acetate. G. H. ELLIS, F. M. STEVENSON, and C. M. CROFT, Assrs. to AMERICAN CELLULOSE AND CHEM. MANUF. CO. (U.S.P. 1,600,277, 21.9.26. Appl., 15.9.24).—See E.P. 224,681; B., 1925, 39.

[Machine for] dyeing knitted and woven fabrics. C. CALLEBAUT and J. DE BLICQUY (E.P. 259,323, 16.7.25).

Tintometer (E.P. 258,366).—See I.

Producing artificial yarns etc. (E.P. 251,580).—See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Equilibrium in the causticising process. I. L. F. GOODWIN (J.S.C.I., 1926, **45**, 360—361 T).—The figures obtained by Lunge for the percentage conversion of sodium carbonate into sodium hydroxide in the lime process are shown to be too high, especially for the more concentrated carbonate solutions. The author's results agree well with those of Le Blanc and Novotny (B., 1906, 1145) and of Walter and Wegscheider (B., 1907, 871). The causticity obtained on treating sodium carbonate solutions with excess of lime is as follows: 2% Na_2CO_3, 99·4% conversion; 4%, 99·2%; 6%, 98·7%; 8%, 97·8%; 10%, 96·5%; 12%, 94·6%; 16%, 89·9%; 20%, 83·5%. A. R. POWELL.

Effect of additions of sodium sulphide on the causticising equilibrium. II. L. F. GOODWIN and I. L. SILLS (J.S.C.I., 1926, **45**, 362—364 T).—The presence of sodium sulphide in sodium carbonate solutions depresses the causticising conversion ratio $NaOH/(NaOH + Na_2CO_3)$ but increases the total causticity, $(NaOH + Na_2S)/(NaOH + Na_2S + Na_2CO_3)$, of the final solution. The results obtained indicate that, under the conditions of the experiment, sodium sulphide is hydrolysed to hydroxide and hydrosulphide to the extent of only about 60%. A. R. POWELL.

Absorption of gases in milk of lime. H. C. WEBER and K. T. NILSSON (Ind. Eng. Chem., 1926, **18**, 1070—1075).—The rate of absorption of carbon dioxide in lime was determined under constant temperature and pressure, but with varying concentrations. Dry calcium oxide and dry calcium hydroxide gave no measurable absorption. With a 3·3% milk of lime the absorption rate was constant within the limits of the experiments. With a 0·15% lime content the rate of absorption was found to be proportional to the distance from saturation. Increase of stirring rate caused an increase in absorption rate. At the highest rate of stirring splashing caused a more rapid increase in absorption. If sodium hydroxide is added to the milk of lime the initial rate of absorption is found to be greater, but the decrease more rapid. On the two-film theory of absorption the effect of the gas film under these conditions is negligible until an accumulation of inert gases, present as impurities in the carbon dioxide, has taken place. C. IRWIN.

Production, properties, and investigation of burnt magnesite. A. STETTBACHER (Chem.-Ztg., 1926, **50**, 741—742, 771—774).—The various stages in the burning of magnesite may be followed by an examination of the refractive index, which increases as the change from carbonate to oxide proceeds. On exposure to air the natural burnt magnesite absorbs moisture and carbon dioxide, but to a much smaller extent than that prepared from precipitated magnesium carbonate. The absorbed water is partly "hygroscopic" and partly "combined" as hydroxide. It is often advisable to burn magnesite at a higher temperature than is actually necessary for decomposition, in order to diminish its hygroscopic character. The higher the temperature of burning the more easily is the setting with magnesium chloride controlled, and the higher is the tensile strength of the cement. Lime is a harmful impurity, but iron and alumina do not influence the formation of the cement. To ascertain the amount of active magnesia, *i.e.*, free MgO, it is necessary to make a complete analysis; but the comparison of the percentages of total magnesia, loss on ignition, and carbon dioxide with similar determinations made immediately after the burning, gives a good idea of the commercial value of the magnesite and the changes it has undergone since burning. Two magnesium oxychloride cements made from the same materials, but from mixtures of different viscosity, had practically the same composition, *e.g.*, loss on ignition 40·8% and 44·1%, $MgCl_2$ 12·7% and 13·7%, moisture 8·8% and 9·9%, combined water 30·1% and 33·6%. F. R. ENNOS.

Apparatus for the direct determination of carbon dioxide. J. E. UNDERWOOD (Ind. Eng. Chem., 1926, **18**, 1069—1070).—The apparatus is designed to reduce the time required for the direct determination of carbon dioxide in limestone and its products while retaining accuracy. Wide absorption tubes are used with a

bulb at the upper end and a stopcock at the lower, and are charged respectively with water and sulphuric acid. They are followed by safety bulbs, charged with zinc in the one case and phosphorus pentoxide in the other. The carbon dioxide freed from hydrogen chloride and water is then absorbed in a Midvale bulb, charged with "Ascarite" and phosphorus pentoxide. Consistent results with limestone and hydrated lime are tabulated.

C. IRWIN.

Manufacture of barium sulphide. W. HIRSCHEL (Chem.-Ztg., 1926, **50**, 692—693).—The use of a gas-fired furnace is recommended for the economical reduction of barium sulphate to sulphide with coal. The primary and secondary air are so regulated that the percentage of carbon dioxide found in the flue gases approximates to the total carbon dioxide, which may be calculated from the rate of combustion of coal in the gas generator and from the rate of reduction of the barium sulphate, in the absence of excess of air, according to the reactions:— $BaSO_4 + 4C = 4CO + BaS$, $4CO + 2O_2 = 4CO_2$.

F. R. ENNOS.

Deliquescent properties of magnesium chloride, of calcium chloride, and of glycerol. E. SWAN (J. Text. Inst., 1926, **17**, T 517—526).—The relative humidity of air at 20° over a range of solutions of magnesium chloride, calcium chloride, and glycerol has been determined. The data are useful to those who may need to control humidity in small vessels. For example, the air above a mixture of magnesium chloride and its saturated solution at 20° remains constant at 34% R.H. as long as crystals and liquid are both present; similarly, a mixture of ammonium nitrate and its saturated solution gives 64·5% R.H. (cf. Prideaux, J.S.C.I., 1920, **39**, 183 T). The work was undertaken, however, to determine the relative values of the two chlorides and glycerol as deliquescents in size, and measurements were made of the hygroscopic properties of dried size film and sized yarn containing known proportions of the materials. The results show that the amount of water taken up by a sized cotton material in an atmosphere of a definite humidity is not simply the sum of the amounts which would have been absorbed by the separate constituents. The addition of a soluble salt to size does not necessarily increase the moisture regain of sized yarn.

J. C. WITHERS.

Influence of added substances on mode of fixation of nitrogen by mixtures of barium carbonate and carbon. P. ASKENASY [with J. BRING] (Z. Elektrochem., 1926, **32**, 216—217; cf. B., 1922, 462).—At 1300° and 1400°, and in presence of 5% of vanadium or 2·5% of barium fluoride, the nitrogen combined is divided between cyanide and cyanamide in the ratio of about 9 : 1; with 5% of iron or nickel the ratio is about 7 : 3. Other metals have varying intermediate effects.

R. CUTHILL.

Determination of silicic acid and fluorine in fluorspar, sand, and silicates. H. DUBIEL (Oesterr. Chem.-Ztg., 1926, **29**, 174—175).—A mixture of fluorspar, sand, and silicates can be conveniently analysed by successive treatment with hydrofluoric acid and concentrated sulphuric acid. A portion of the finely-powdered material is dried at 250° (glowing must be avoided) in a platinum vessel, hydrofluoric acid is added, the mixture heated to dryness, and finally to 250°, and the process repeated until constant weight is reached. The loss of weight gives the content of silicic acid. The residue is repeatedly treated with concentrated sulphuric acid, heated to dryness, and finally to 250°, until constant weight is reached, the calcium and other basic constituents being thereby converted into sulphates. The increase of weight thus obtained + the already calculated silicic acid × the factor 38/58 = the fluorine content. Metallic oxides are determined in the residue by digesting with hot, moderately dilute hydrochloric acid. The undissolved gypsum, filtered and washed, together with any calcium precipitated (as oxalate) from the filtrate, is converted into and determined as sulphate by treatment as above with sulphuric acid. Any remaining basic constituents are determined in the hydrochloric filtrate in the usual way. A modification is necessary if the original mixture contains calcium carbonate. After the first drying at 250°, the material is heated to dryness with 10% acetic acid, the residue (calcium acetate) being washed with distilled water, and added, together with the incinerated filter, to the crucible.

P. V. MCKIE.

Sodium perborate. SBORGI and LENZI.—See XI.

Action of citric acid solutions on phosphates. ANDREASEN.—See XVI.

Lime-sulphur-calcium arsenate spray. GOODWIN and MARTIN.—See XVI.

Solubility of the copper in basic copper carbonate. HALL.—See XIX.

Colorimetric determination of iron [in tea etc.] by the ferrocyanide method. MUMMERY.—See XIX.

PATENTS.

Treating soda solutions contaminated with cellulose-like substances. LA SOIE D'AUBENTON CO. (E.P. 249,091, 19.2.26. Conv., 11.3.25).—Caustic liquors containing cellulose products from the pressing operation in the manufacture of artificial silk by the viscose process are treated with carbon dioxide at the ordinary temperature, whereby the cellulose products are quantitatively precipitated, and are separated by decantation, filtration, or centrifuging, while the sodium bicarbonate formed is converted into carbonate, and is causticised for further use.

W. G. CAREY.

Manufacture of ammonia. H. W. BLACKBURN and W. THOMAS (E.P. 257,689 and 258,154, 9.6.25).—(A) Pure nitrogen and steam, in the proportion of 1 pt. of nitrogen by weight to 4 pts. of steam, are passed over a heated catalyst at a pressure of about 2 atm. The catalyst may be iron, for which the optimum reaction temperature is 500°, iron in presence of nickel at a temperature of 1000°, nickel oxide at 400°, or ferrous oxide at 500°. (B) A mixture of pure nitrogen and steam in the ratio of 1 pt. of nitrogen by weight to 4 pts. of steam is brought into contact with heated charcoal at a pressure of about 2 atm. The temperature may be between 450° and 1300°, but is preferably about 800°. The charcoal appears to act as a catalyst, and undergoes no permanent change.

W. G. CAREY.

Synthesis of ammonia. F. Uhde (E.P. 247,226, 8.2.26. Conv., 9.2.25).—Increased activity of the catalyst is obtained by using a solution of an alkali or alkaline earth metal in molten alkali amides; in such cases both metal and solvent act as cleansers for the gas, and the gaseous mixture of nitrogen and hydrogen may be passed through the catalyst at 200—250°.
F. G. Crosse.

Apparatus for the production of synthetic ammonia. "Montecatini," Soc. Gen. per l'Ind. Min. ed Agric., and G. Fauser (E.P. 240,436, 8.9.25. Conv., 24.9.24).—An apparatus is described consisting of two steel cylinders connected together, the lower one containing a bundle of longitudinal heat exchange tubes, and the upper one another bundle of tubes, also longitudinal, and in the form of a truncated cone, a heat insulation lining surrounding the latter tubes to protect the outer cylinder, and a reaction chamber arranged inside the bundle surrounded by a refractory material with a thermo-electric coil between. The mixture of nitrogen and hydrogen is passed, under pressure, round the lower tubes, thereby becoming heated. This pre-heated mixture passes to the upper tubes, and there comes into contact with the thermo-electric coil, which raises it to its reaction temperature. F. G. Crosse.

Apparatus for the synthesis of ammonia. G. Cicali (E.P. 258,340, 17.6.25).—The apparatus comprises an external cylinder with heat-resisting lining and three co-axial cylinders, by means of which the cold mixture which is to be subjected to catalysis is divided into two cold currents, one being conveyed direct on to the outer wall of the cylinder containing the catalyst, the other passing over and cooling the pressure-resisting walls, exchanging heat with a portion only of the hot gases leaving the reaction chamber, finally uniting with the first current. The united currents then absorb heat from the catalyst cylinder, the gases leaving the reaction chamber being divided also into two currents, one leaving the apparatus without exchange of heat, whilst the other exchanges heat with the second cold current. The whole of the ammonia is condensed in two condensers, one of which is water-cooled, the other cooled by the liquid ammonia condensed in both condensers.
H. Royal-Dawson.

Production of solid compounds of ammonia, carbon dioxide, and water. Synthetic Ammonia & Nitrates, Ltd., and T. Coxon (E.P. 258,048, 23.7.25).—Ammonia gas and carbon dioxide are pumped at atmospheric pressure into water at 20—50° until the concentration of each gas is 30% by weight of the total solution. On precipitation of ammonium carbamate, the ammonia and carbon dioxide are supplied in the proportion of two parts of the former to one of the latter, thus establishing a continuous production of the carbamate salt, which is then separated from the solution together with some adherent mother liquor, the moist crystals being treated with carbon dioxide and extra water (as steam) until a final dry product remains. H. Royal-Dawson.

Manufacture of finely divided metal oxides and silica. J. Y. Johnson. From Badische Anilin & Soda-Fabrik (E.P. 258,313, 15.5.25).—Iron or other metal carbonyl, or a volatile halide, is thoroughly mixed with air or a combustible liquid, *e.g.*, alcohol, and forced through a nozzle into a spray by means of air or oxygen, and ignited by means of platinised asbestos, the resulting metallic oxides being then separated from the products of combustion. H. Royal-Dawson.

Manufacture of pure hydrochloric acid. Verein für Chem. & Metallurg. Prod. (E.P. 248,738, 25.2.26. Conv., 9.3.25).—Gaseous hydrochloric acid is treated with chlorine sufficient to oxidise the impurities, then filtered through activated carbon. H. Royal-Dawson.

Manufacture of hydrochloric acid. D. Tyrer (E.P. 259,348, 19.8.25).—A mixture of ferrous chloride with an iron compound, *e.g.*, yellow hydrated oxide or hydrated ferrous oxide, is decomposed at a suitable temperature (350—450°) by a current of air or oxygen and steam. The ferrous chloride solution—a by-product of galvanising baths—is evaporated down with a finely-divided iron oxide, and the granular mixture exposed in thin layers to the oxidising atmosphere, the amount of water vapour being so adjusted that acid of a commercial strength is produced. F. G. Crosse.

Treating residue from the manufacture of acetylene for the recovery of lime products. A. Stephenson, and Allen-Liversidge, Ltd. (E.P. 258,660, 24.6.25; cf. following abstract).—Quick lime is added to the residue and the mixture agitated in an apparatus adapted for de-watering during transit to the screening apparatus, the larger products from which are returned for further treatment with or without crushing.
H. Royal-Dawson.

Production of blanc fixe from barium sulphide. A. Jahl (E.P. 259,102, 25.2.26. Conv., 5.11.25).—An alkali sulphate is added to an aqueous solution of barium sulphide containing relatively more hydrosulphide than hydroxide, formed by the addition of the requisite amount of an acid or acid salt to convert 10—15% of sulphide into hydrosulphide. H. Royal-Dawson.

Production of soluble fluorides. A. L. Mond. From Chem. Fabr. Greisheim-Elektron (E.P. 257,981, 4.6.25).—The process consists in heating fluorspar with hydrochloric acid in the presence of silicic acid and barium or sodium fluoride. Sparingly soluble silicofluorides are produced which are filtered and decomposed by means of ammonia or potash, giving easily soluble fluorides and a residue which is used again in the first part of the process. H. Royal-Dawson.

Production of anhydrous magnesium chloride. A. L. Mond. From I.-G. Farbenind. A.-G. (E.P. 259,498, 13.5.26).—A self-binding mixture of oxidic compounds, molten hydrates of magnesium chloride, or magnesium chloride lyes, with carbon or sawdust is made which, at temperatures below the melting-point of magnesium chloride, forms a coke and occupies a large space, giving the necessary porosity for the reactions, and leaving sufficient space for its increase in volume on conversion by chlorination into solid magnesium chloride. An example is given in which 200 kg. of $MgCl_2,6H_2O$ are fused, and to the melt is added 40 kg. of ground burnt magnesite with 18 kg. of carbon. The mixture swells and solidifies. After preliminary drying it is broken up and chlorinated at 500—550°. A mixture

of caustic burnt magnesite, charcoal, and sawdust, saturated with magnesium chloride lye (about d 1·18), may be used. This is granulated, dried at 350°, and treated as before. F. G. CROSSE.

Manufacture of alumina salts from alumina-silicate and minerals containing alumina. O. LEDERER and W. STANCZAK (E.P. 246,512, 23.1.26. Conv., 24.1.25).—Clay or other aluminous material is boiled with a concentrated solution of sodium carbonate, under 14 atm. pressure, at about 200° for 1½ hrs. After refrigeration the filtered solution of sodium carbonate is concentrated and re-used, the residue being rendered easily soluble in acids. A sample of blue clay of Wildstein containing 34·41% Al_2O_3 and 50·03% SiO_2 yielded 98·45% of its alumina content in a pure state. F. G. CROSSE.

Manufacture of aluminium chloride. R. J. DEARBORN, Assr. to the TEXAS Co. (U.S.P. 1,600,216, 21.9.26. Appl., 26.2.25).—A mixture of aluminium ore and carbonaceous material is simultaneously coked and purified by heating and chlorinating at a relatively low temperature, and the mass is further chlorinated at relatively high temperature without loss of heat. H. ROYAL-DAWSON.

Cyanide product. K. F. COOPER, Assr. to AMER. CYANAMID Co. (U.S.P. 1,599,212, 7.9.26. Appl., 29.5.23). —Crude cyanide containing decomposition products is improved by fusion with ferrocyanide, and removing any iron that may separate out during the process, the iron-free product being then cooled and recovered. H. ROYAL-DAWSON.

Performing chemical reactions (E.P. 231,901).—See I.

VIII.—GLASS; CERAMICS.

Premature failure of combustion chamber material. A. J. DALE (Gas J., 1926, **175**, 862—865).—A highly siliceous refractory material which had failed in use in the combustion chamber of a gas retort setting was examined according to the Mellor–Moore load test. Samples of the unused material were heated at a fixed rate under loads of 50, 10, and 4 lb./sq. in. respectively, and the deformation of the sample was plotted against temperature. In all cases the expansion ceased at about 1000°, showing that a uniformly distributed portion of the material had become plastic. Under loads of 10 and 50 lb./sq. in. the specimen collapsed at 1100° and 1150°, respectively. Under 4 lb./sq. in. the rate of subsidence decreased between 1150° and 1320°, after which the specimen collapsed. This diminished rate is due to the specimen yielding to internal stresses caused by expansion during the conversion of quartz to cristobalite. The causes of failure in use were twofold: firstly, the premature fusion of the bond and the lack of cohesion of the bond in the viscous state with the siliceous grains, and, secondly, internal stresses due to the rapid expansion of unconverted quartz. S. PEXTON.

PATENTS.

Apparatus for melting and fining glass. R. D. PIKE, Assr. to COLE–FRENCH Co. (U.S.P. 1,598,307, 31.8.26. Appl., 25.10.22).—The apparatus consists of an open-hearth regenerative furnace, the dimension of which with the flame is over twice as great as that transverse to the flame. The raw mix is fed through small bays into the furnace and leaves the latter by a submerged port, the refractories surrounding which are protected by a water jacket. The glass passes through a fining compartment, pursuing a tortuous up and down path, the temperature being maintained by a flame counter current to the glass. The fined glass then enters a working compartment, whence it may be delivered as desired. R. B. CLARKE.

Fining glass. R. D. PIKE, Assr. to COLE–FRENCH Co. (U.S.P. 1,598,308, 31.8.26. Appl., 1.11.22).—To facilitate removal of the gaseous products of decomposition produced in glass fining, the glass on leaving the melting furnace passes through a vacuum chamber wherein decreased pressure causes the dissolved and absorbed gases to be liberated. The temperature of the glass while passing through the chamber is maintained electrically. R. B. CLARKE.

Glass furnace. C. D. MCARTHUR, Assr. to BLAW-KNOX Co. (U.S.P. 1,598,779, 7.9.26. Appl., 11.3.21).—A glass tank furnace is provided with a bottom formed of refractory blocks seated upon a series of hollow water-cooled members, which are independent of each other and separately removable. B. W. CLARKE.

Ceramic safety tile. M. C. BOOZE, Assr. to NORTON Co. (U.S.P. 1,600,925, 21.9.26. Appl., 27.10.22).—A safety tile is described consisting of wear-resisting, abrasive, anti-slipping grains of porcelain bonded into an integral non-homogeneous highly porous mass by a vitrified ceramic bond, which is softer and more vitreous at the firing temperature than the grains, and forms 25—60% by weight of the total mass. B. W. CLARKE.

IX.—BUILDING MATERIALS.

Silica–lime bricks. R. DE FAZI (Annali Chim. Appl., 1926, **16**, 400—404).—Bricks with a compressive strength of 200 kg./sq. cm. may be made by mixing 85% of Ostia sand with 15% of lime and simultaneously quenching the lime with steam at about 8 atm. pressure; the pressed bricks are kept for 10 hours in an autoclave at a pressure of 8 atm. When subjected to a temperature of −5° for periods varying from 10 to 40 days, such bricks undergo no physical or chemical changes adversely affecting the strength, which in most cases exhibits appreciable increase. T. H. POPE.

Determination of free lime in hydraulic cement. F. L. BRADY and F. J. MCCONNELL (Dept. Sci. Ind. Res., Building Res., Tech. Paper No. 4, 1926, 1—13).—"Wet" methods of determining the free lime in hydraulic cements are not in general so satisfactory as "dry" methods, owing to the fact that further hydration proceeds during the determinations and produces more free lime. Extraction with a 20% sugar solution gives useful information for set cements, and is a rapid method for testing where only small quantities of material are available. Passow's method (see Zement, 1923, **12**, 87) in which ammonium carbonate is heated with the cement and forms calcium carbonate with the free lime, is not very satisfactory owing to hydration effects, ammonium carbamate giving better results. The most

satisfactory method for determining free lime is a thermal method, in which the cement is heated at 900° for ten minutes, converting the calcium hydroxide into calcium oxide, which is then determined by the rise in temperature produced when it is mixed with water. This gives reliable results for set and unset cements, and is especially valuable in investigating the fire-resisting properties of cement or concrete, which depend apparently on the free lime content, and also in determining the degree of aeration of the cement. B. W. Clarke.

Burnt magnesite. Stettbacher.—See VII.

Stability of blast furnace slag and cause of its disintegration. Guttmann.—See X.

Patent.

Hydration of concrete and other similar materials. F. W. V. Fitzgerald (E.P. 258,320, 9.6.25).—Cement or plaster is mixed with materials which liberate water when subjected to heat or pressure, such as salts containing water of crystallisation, the gelatinous precipitate formed by adding ammonium carbonate to a solution of alum, a solidified solution of sodium or potassium silicate or the like, and the mixture pressed into moulds at a temperature of about 100°. Bitumen, wax, stearine, colouring and filling materials may be added if desired. B. W. Clarke.

Manufacture of cementitious substances. P. Budnikov (E.P. 258,727, 7.9.25).—Finely ground over-burnt gypsum or anhydrite is mixed with a "catalytic" agent such as a mixture of a bisulphate of an alkali metal with copper sulphate or ferrous sulphate, with or without the addition of iron filings, calcium phosphate, powdered glass, organic matter such as peat, and asbestos. B. W. Clarke.

Making cement from town refuse. G. E. Heyl (E.P. 259,503, 31.5.26).—Refuse is calcined in a rotary kiln at a temperature not exceeding the fusion temperature of the ash, with the addition of suitable fuel, by means of hot combustion gases from another furnace. The product is ground and mixed with the necessary calcareous and aluminous materials to form a raw mixture suitable for making Portland cement, which is calcined in a rotary kiln in the usual manner. B. W. Clarke.

Manufacturing artificial stone. F. R. A. Sundell (E.P. 239,497, 5.8.25. Conv., 6.9.24).—Glue is dissolved in water to the extent of 1 pt. in 40—70 pts., and 8—12 pts. by weight of this solution are mixed with 1 pt. of a suspension of lime in water (1—4 pts. by weight of lime in 3 pts. of water), gypsum being added in sufficient quantity to produce a mass of the required consistency for artificial stone. This is moulded on a glass plate if a polished surface is required, and subsequently loosened by the application of hot ashes to the back of the plate. B. W. Clarke.

Manufacturing porous artificial stone. J. A. Eriksson (E.P. 258,073, 31.8.25).—A finely divided intimate mixture of lime and a siliceous material, *e.g.*, calcined slate (alum shale ashes), is mixed with powdered aluminium or zinc, which generates gas when the mass is treated with water. After gauging with water, with or without the addition of Portland cement, the porous mass is subjected to the action of steam under pressure, thereby producing, after 5—10 hours' hardening, a strong and durable porous artificial stone (gas concrete). B. W. Clarke.

Supplying raw slurry to rotary cement-burning kilns. N. Nielsen (E.P. 258,199, 4.5.26).—Raw slurry is subjected to a screening process before being supplied to the atomising nozzle of a rotary kiln. The cement dust, usually carried over by the smoke from the end of the kiln, is collected in a suitable dust chamber, elevated by buckets, and allowed to return to the kiln by gravity, or, alternatively, admixed with a small quantity of air so that it can be pumped back to the kiln, or blown into the kiln with the powdered coal used for burning. This obviates waste of heat in redrying the collected dust, which by present methods is returned in a wet state. A dust filter consisting of chains kept wet by sprinkling with water or slurry is preferably provided in the drying zone of the kiln, and pressure-regulating devices are provided with the feed pipe from the raw slurry tank to prevent damage to the nozzle if choking occurs. B. W. Clarke.

Improving the properties of wood. W. H. Kobbé, Assr. to Texas Gulf Sulphur Co. (U.S.P. 1,599,135, 7.9.26. Appl., 24.11.25).—Wood in its natural state is immersed in a bath of sulphur at a temperature of 140—150°, until practically the whole of the moisture has been expelled from the pores of the wood. The temperature of the bath is then reduced to about the melting point of sulphur, and maintained at this temperature until the sulphur has sufficiently penetrated the pores, the sulphur finally being allowed to congeal within the pores. B. W. Clarke.

Prevention of sap staining and molding [on wood]. E. Bateman and E. E. Hubert (U.S.P. 1,598,699, 7.9.26. Appl., 23.5.24).—The process consists in impregnating the wooden products with an aqueous solution of an alkali phenolate which will react with the air forming an alkali carbonate and free phenol, which is dissipated by the air as the water evaporates. F. G. Crosse.

Materials for coating walls. O. Kunze (E.P. 258,164, 26.2.26).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Influence of materials and operation duration on the heat equilibrium and temperature in the Thomas process. H. Bansen (Stahl u. Eisen, 1926, 46, 1277—1283).—Each constituent of a Thomas converter charge is considered in relation to its heat contribution to the thermal equilibrium and to its effect on the temperature produced. Variation of efficiency with time of operation is also considered. L. M. Clark.

Graphitisation at constant temperature. H. A. Swartz (Trans. Amer. Soc. Steel Treat., 1926, 9, 883—906).—Four typical graphitisation curves at a temperature above A1 are given for irons containing 2·25—2·60% C, the ordinates representing the degree of completion of graphitisation. The curves can be brought into coincidence by shifting them horizontally.

Photomicrographs show that graphitisation proceeds by an increase in size rather than in number of carbon nodules which are surrounded by solid solution lower in carbon content than the austenite in which the nuclei are formed. A mathematical analysis is made of the data of graphitisation, and it is shown that the rate of graphitisation is determined by the rate of migration of carbon in some form through solid solution or ferrite. It is concluded that the graphitisation rate is proportional to an experimental constant which provides a valuable means of determination of this rate. Either carbon in all its forms migrates in iron at the same rate, or the form in which it migrates is always the same or changes continuously with a corresponding change in the graphitising rate. The logarithm of the rate of graphitisation is proportional to the temperature. Equal increments of silicon multiply the graphitisation rates by equal amounts, except as regards low silicon contents. Silicon also decreases the thermal coefficient of the velocity of graphitisation. The *modus operandi* of the process of graphitisation is discussed.

T. H. Burnham.

Irregular carburisation of iron and iron alloys—the cause and prevention. W. J. Merten (Trans. Amer. Soc. Steel Treat., 1926, **9**, 907—928).—The conditions for consistently uniform carburisation are discussed. The mixture should not dissociate until the critical point has been passed, and the residue should be regenerative. The steel parts should have clean surfaces free from matter chemically active below or at the carburising temperature. They should possess a uniform grain structure produced by normalising or annealing prior to treatment. The articles should be so arranged as to expose all parts uniformly to the carburising gas. A self-sealing box with copper gaskets to maintain a positive pressure is recommended. For partial or selective cementation the use of solid copper protectors is preferred. For carburising by purely gas contact, a set of copper screens were used which prevented the compound from coming into contact with the parts. By this means the uniformity and depth of case were improved.

T. H. Burnham.

Progressive carburisation in rotary electric furnaces. H. E. Martin (Trans. Amer. Soc. Steel Treat., 1926, **9**, 933—937).—Experiments on firing heat-treatment furnaces with city gas, producer gas, oil, and electricity resulted in the adoption of the last-named on account of greater control and flexibility of heat input, improved working conditions, and greater output. For case hardening, three rotary 500-kw. furnaces were erected of 26 ft. $3\frac{1}{2}$ in. outside diam., the total loaded weight of the rotary tables being 60—70 tons. The table speed was $10\frac{1}{2}$—$15\frac{1}{3}$ hrs./rev. The heating elements were 80—20 nickel-chromium ribbon, the current density being 8 watts/sq. in. The carburisation was uniform with practically no rejections. The process was continuous, over 7·7 million lb. of material being treated each year at approximately 0·5 kw./lb. power consumption.

T. H. Burnham.

Iron and steel: sulphuric and nitric [acid] corrosion. S. C. Bate (Chem. Age, 1926, **15**, 419—420).—Oleum and strong (100%) sulphuric acid dissolve steel somewhat more readily than iron, whereas 65—95% sulphuric acid dissolves iron slightly more readily than steel, and weaker acid iron much more rapidly than steel. Nitric acid solutions stronger than 65% have a very energetic action on iron, but only a slight action on steel; weaker solutions, however, dissolve both metals readily. Mixtures of sulphuric and nitric acids containing up to 10% of the latter react readily with iron, and the rate of action is increased by the addition of water; steel, on the other hand, is only slightly affected when less than 25% of water is present. Acid mixtures containing more than 10% of nitric acid exert the minimum corrosive action on iron when 20% of water is present, and on steel when 15% of water is present. A protective action is afforded by the addition of 30—35% of nitric acid to sulphuric acid–water mixtures, but the presence of nitrous acid has the opposite effect, and causes rapid dissolution of both iron and steel in acid mixtures. Prolonged exposure of iron or steel to the action of acids of such concentration that corrosion is fairly slow results in an appreciable retardation of the rate of dissolution of the metal after a certain period owing to the formation of a protective coating on its surface. If this coating is removed the subsequent rate of corrosion is still somewhat less than the original rate, and is also more uniform.

A. R. Powell.

Transformations in hardened steel during tempering. H. Hanemann and L. Traeger (Stahl u. Eisen, 1926, **46**, 1508—1514).—Prolonged tempering of quenched steel at temperatures between 50° and 450° results in three changes in the structure and properties of the metal. The first change begins at 75°, and is complete in 14 hrs. at 100°; the metal contracts 0·12% when 0·97% C is present, and the hardness suffers a sudden slight fall, whilst a change takes place in the martensite needles, causing them to be more readily attacked by etching reagents. On further heating to 235° a dilatation of 0·035% takes place in a 0·97% C steel, and the hardness falls somewhat more than at the previous transformation point. As this second change does not occur in steels containing less than 0·8% C, it appears to be due to the decomposition of the austenite phase. The third transformation occurs at 300°, and is accompanied by a contraction of 0·18% and a sudden fall in hardness, but by no visible structural change. Above 400°, however, the usual sorbitic structure appears. Reasons are advanced for assuming that the transformation at 100° is accompanied by the formation of a new phase, which the authors designate as the ζ phase and suggest that it consists of a new carbide characterised by ready solubility in acid, a smaller electrical resistance and hardness than martensite and a greater hardness than the heterogeneous cementite–α-iron phase; in the microstructure after etching with nitric acid the ζ constituent appears as black needles. This constituent seems to be identical with the α-martensite described by Honda, but, contrary to the latter's observations, it can not be produced directly by quenching austenitic steel in oil at 200°. Once the ζ-phase has been produced by tempering at 100° the steel suffers no further volume change below 200°. The usual tempering temperature of steel is between 200° and 300°, but the period of tempering is

never sufficiently prolonged to obtain even approximate equilibrium between the various phases, so that if it is desired to obtain a tempered steel which will not suffer any volume change at temperatures above 280° it must be tempered at 400° to obtain the α-iron–cementite structure. A. R. POWELL.

Influence of silicon on the magnetic properties of steel. K. DAEVES (Z. Elektrochem., 1926, **32**, 479—481).—Mainly a discussion of previous work (cf. B., 1924, 983) with reference to possible reduction of power losses in transformers and dynamos. With steel containing more than 1·3% of silicon the α and δ forms of iron are interconvertible without the intermediate formation of γ-iron, so that the grain size in the cold metal is dependent only on the extent of working and the annealing temperature. A coarse-grained metal is consequently obtainable, and this is advantageous, since, for silicon steel of low carbon content or with most of the carbon converted into graphite by heating at about 800°, the power loss decreases nearly linearly with increase of grain size. Theories of the effect of grain size on power losses are discussed. H. J. T. ELLINGHAM.

Determination of sulphur in iron. K. K. JÄRVINEN (Z. anal. Chem., 1926, **68**, 397—404).—A long series of tests has been carried out to determine the quantitative relative effects of the various sources of error, and the following rapid and accurate method has been worked out: 5 g. of the filings are treated slowly in presence of 100 c.c. of water with 8—9 c.c. of bromine, the excess is removed by boiling, and 20—30 c.c. of 3*N*-barium chloride, with water to make 100—150 c.c., are added after cooling. After 3 hrs. the precipitate is filtered, washed, and ignited, and the residue fused with sodium potassium carbonate and a little sodium nitrate. The melt is dissolved, and the filtered solution acidified with hydrochloric acid, heated, and treated with dilute barium chloride, a few drops of lacmoid or Congo-red being added to prevent the barium sulphate passing through the filter. S. I. LEVY.

Determination of gases in iron and steel. P. KLINGER (Stahl u. Eisen, 1926, **46**, 1245—1254, 1284—1288, 1353—1356; cf. B., 1925, 993).—Methods for determining the amounts of gas mechanically contained or dissolved in iron or steel are critically reviewed. The total amount of gas is obtained either by heating the material *in vacuo* with or without a flux or by dissolving it in a suitable reagent. The results from basic Siemens–Martin steels of varying carbon content depend on the method used, treatment of the sample *in vacuo* giving the greatest amount of gas. The production of carbon monoxide by reaction of cementite or graphite with oxides of iron, manganese, silicon, and aluminium is investigated, and it is considered that both carbon monoxide and carbon dioxide obtained in vacuum extractions of steels or on dissolving in chemical reagents are products of such reactions, and are not in solution in the steel. The amount of these reaction gases provides an index to the amount of oxygen present in the sample. The most trustworthy method for the determination of nitrogen and hydrogen is considered to be fusion of the sample with a tin–antimony flux, whereby iron nitride is completely decomposed. The formation of blowholes in steel is ascribed to liberation of carbon monoxide as a reaction product. This gas carries with it from the fused metal some hydrogen and nitrogen, which remain in the blowhole when the monoxide is decomposed at the wall of the cavity. L. M. CLARK.

Testing the stability of blast-furnace slag by means of ultra-violet light and the cause of the disintegration of the slag. A. GUTTMANN (Stahl u. Eisen, 1926, **46**, 1423—1428).—On exposing pieces of iron blast-furnace slags to ultra-violet light, characteristic fluorescence colours are produced which serve to distinguish stable slags from those which will disintegrate on keeping. The former show on freshly fractured surfaces an evenly distributed dark to light violet fluorescence, and on surfaces that have been exposed to the weather yellowish-white spots on a violet background. Unstable slags, on the other hand, show numerous yellow to red or cinnamon-brown spots often gathered in large clusters on a violet background. Dark violet spots on a nearly black background are simply reflexion phenomena from the crystal faces. From the examination of numerous slags in this way, as well as under the polarising microscope, it is shown that the cause of the disintegration of unstable slags is the slow transformation of α- and β-dicalcium silicate into the γ modification which is associated with an increase of 10% in the volume of this compound, and that the γ-form is the cause of the red or brown spots mentioned above. The formation of unstable slags may be avoided by reducing the lime content of the charge or by substituting a certain amount of magnesia or other bases for part of the lime; too high a content of ferrous oxide is inadvisable, as it results in a slag which decomposes somewhat readily in moist air. A. R. POWELL.

Transformations of aluminium-bronzes. J. BOULDOIRES (Compt. rend., 1926, **183**, 660—661).—Transformation points are recorded for an aluminium-bronze containing 87% Cu, which was heated to 850° and allowed to cool slowly in 15 hrs., at 515°, 560°, and 690° during the heating period, and at 650°, 510°, and 480° during the cooling period. When this sample is reheated to 530° the first transformation does not take place. If cooling takes place quickly in a jet of air, the transformation points are found at 635,° 500°, and 460°. The resistivity is also affected by the rate of cooling. In the cases of tempered samples heated for a second time to various temperatures, maximum values of resistivity are recorded for 500°. At lower temperatures the reheating has the least effect. Micrographical observations indicate that acicular structures correspond with the maximum resistivity, since they are brought about by the same conditions of tempering and reheating. J. GRANT.

Influence of chemical composition of alloys on the power of obtaining moulded pieces (or flowability). L. GUILLET and A. PORTEVIN (Compt. rend., 1926, **183**, 634—636).—The authors distinguish between the fluidity of an alloy and its power of filling a mould completely (flowability). The latter has been determined at 550° and 450° for alloys of tin with bismuth and with lead, by measuring the length of a horizontal spiral-shaped channel filled by the liquid alloy. The

curves obtained are of the same form, maximum values being obtained at the eutectic points. The latter phenomenon is due to the low m.p. of the alloy, and it is suggested that the "flowability" varies as a continuous function of the excess temperature of the melt over that at which solidification starts. The speed of solidification is a specific property of the alloy, and must also be taken into account. J. GRANT.

PATENTS.

Steel alloys [for tools]. GELSENKIRCHENER GUSSSTAHL U. EISENWERKE A.-G., ABT. STAHLWERKE KRIEGER, and B. RASCHKE (E.P. 250,560, 26.3.26. Conv., 11.4.25). —A durable steel for rolling mill mandrels is suggested having, in addition to the usual small traces of phosphorus and sulphur, the following composition: 0·33% C, 0·16% Si, 0·20% Mn, 0·46% Cr, 1·69% Mo, 4·18% Ni. F. G. CROSSE.

Increasing the fluidity of molten metal [iron and steel]. A. PACZ (U.S.P. 1,596,888, 24.8.26. Appl., 7.11.22).—Molten cast iron or steel, which has cooled too much in pouring, can be rendered sufficiently fluid to complete the operation by adding a mixture of powdered aluminium, boric, and ferric oxides. The heat evolved by the ensuing "thermite" reaction raises the temperature of the metal, the fluidity of which is also increased by the formation of an alloy of iron and boron which has a relatively low m.p. The presence of boron is not injurious, but increases the tensile strength of the product. The addition of boric oxide to the mixtures employed in thermite welding, and, mixed with thermite as above, to other molten metals, is also of advantage. T. S. WHEELER.

Blast furnace. J. KENNEDY (U.S.P. 1,598,777, 7.9.26. Appl., 2.7.25).—A blast furnace is described consisting of a small and a large bell, the former being laterally adjustable and acting as a guide for a rod supporting the large bell. B. W. CLARKE.

Production and treatment of refractory metals [chromium]. A. S. CACHEMAILLE. From WESTINGHOUSE LAMP CO. (E.P. 258,024, 19.6.25).—Powdered pure chromium is agglomerated into a bar and slowly heated in a high vacuum, so as to sinter the particles into a compact mass. The bar is then further heated to just below the fusing temperature by electro-magnetic induction, and after cooling to redness the metal may be hammered, rolled, or drawn until the desired ductility is obtained. C. A. KING.

Extracting volatile metal from ore. E. M. JOHNSON (E.P. 258,948, 1.7.25).—In conjunction with a series of horizontal retorts arranged both laterally and vertically, and adapted for the volatilisation of metals, travelling cars, carrying a charge of ore in troughs corresponding in position with that of the retorts, are caused to register with both ends of the retort bench and charge the retorts simultaneously. C. A. KING.

Copper-silicon alloys. W. DENECKE (E.P. 259,091, 2.2.26).—Resistant alloys for use in chemical industry contain Si 5—10%; Fe, Ni, Co (separately or together) 1·5—12%; the remainder being copper. The alloys may also contain up to 5% of tungsten, molybdenum, titanium, or vanadium, and up to 1% of arsenic. C. A. KING.

Alloy [for printing rollers]. J. WALRATH (E.P. 258,490, 15.3.26).—An alloy suitable for the production of printing rollers and blocks used in the manufacture of wall paper contains Al 84%, Sn 10%, Zn 6%. C. A. KING.

Silver alloy. SOC. FRANÇ. DE MONNAYAGE (E.P. 259,108, 5.3.26. Conv., 2.2.26).—A homogeneous silver alloy, of white colour, suitable for goldsmiths' work, contains 30—70% of silver, also nickel, copper, and zinc, the last being wholly or partly replaceable by cadmium. Two typical alloys have the compositions Ag 40% (50), Ni 12% (10), Cu 35% (30), Zn 13% (10). In order to prevent volatilisation of zinc during the preparation of an alloy, a silver-zinc alloy of correct composition is first prepared by heating a mixture of finely-divided zinc and silver at a comparatively slow rate, so that a continuous formation of intermediate alloys occurs from about 420°. C. A. KING.

Flux materials for use in arc welding and methods of coating electrodes. HUME STEEL, LTD., and W. R. HUME (E.P. 258,036, 6.7.25).—Electrodes for use in arc welding are coated with potassium dichromate. A suitable method of preparing such an electrode is by dipping the red-hot electrode wire in a mixture of powdered potassium dichromate and a refractory material, *e.g.*, asbestos. C. A. KING.

Electrode for welding and like purposes. H. D. LLOYD and C. E. HILL, Assrs. to THE WHITECROSS CO., LTD. (U.S.P. 1,599,056, 7.9.26. Appl., 12.11.23. Conv., 18.12.22).—A coating composition for electrodes contains siliceous fireclay and titaniferous iron ore, and is substantially free from carbonates and carbon. J. S. G. THOMAS.

Electrode for use in electric arc welding and the cutting of metals. THE QUASI-ARC CO., LTD., and A. P. STROHMENGER (E.P. 259,365, 16.9.25).—A metallic core is provided with a yarn or fibre covering having as a substantial constituent a fibrous silicate, *e.g.*, amosite, or a mixture of amosite and crocidolite, the iron content of which exceeds 30%. J. S. G. THOMAS.

Metallising fine ore concentrates. D. M. CRIST, Assr. to TRIUMPH STEEL CO. (U.S.P. 1,595,454, 10.8.26. Appl., 26.12.24).—The ground ore is mixed with carbonaceous material and passed continuously through a zone where it is heated out of contact with air to such a temperature and for such a time as to become completely reduced without fusion. The gas evolved is burnt under the furnace. E. S. KREIS.

Protecting metallic articles from oxidation, deterioration, or corrosion. METALLISATION, LTD., and W. E. BALLARD (E.P. 259,289, 4.7.25).—The method consists in forming on the articles, cleaned by sand-blasting, a coating 0·005—0·006 in. thick of aluminium by means of the Schoop metal-spraying process, which is then covered with a carbonaceous material, *e.g.*, lamp black or bitumastic paint, and heated to 650°. F. G. CROSSE.

See also Class XI (following): **Electrolytic deposition of chromium** (E.P. 258,724 and 259,118); **Extraction of tin from alloys containing lead** (E.P. 240,147); **Electrolytic deposition of metallic coatings on metals** (E.P. 259,307); **Cleaning silver etc.** (E.P. 258,726).

XI.—ELECTROTECHNICS.

Electrolytic preparation of sodium perborate. U. Sborgi and D. Lenzi (Giorn. Chim. Ind. Appl., 1926, 8, 423—427).—A mixture containing 23% of crystallised sodium carbonate, 3·3% of borax, 1·5% of sodium bicarbonate, and 10·5% of chromic oxide, together with a small quantity of salt, is electrolysed, using a gilded copper sheet anode and an iron cathode. Further powdered borax and carbonate are added during the reaction. With a current of 20—25 amp. (10—12 volts) a solid, well-crystallised perborate, of 99—100% purity, representing a yield of 35—40% on the current used, is obtained. At intervals of 24 hrs. the reaction is stopped, and the mixture centrifuged and washed rapidly with cold water. The filtrate can then be used for further electrolysis. P. V. McKie.

Patents.

Building and starting electric induction furnaces. C. A. Brayton, jun., Assr. to Induction Furnace Co. (U.S.P. 1,598,236, 31.8.26. Appl., 24.9.20).—A refractory lining is packed about a short-circuited metallic core, a casing for which is provided in which the cross-sectional air space is sufficient to permit lateral displacement of the core during expansion, thus forming a furnace channel. A low current is induced in the core to heat it and dry out the lining, the current being then increased to melt the core. L. M. Clark.

High-frequency electric furnace. C. Lorenz A.-G. (E.P. 249,843, 11.3.26. Conv., 30.3.25).—In a high-frequency electric furnace in which alternating current is passed through a heating coil outside the crucible, and metal contained in the crucible is heated by the induced eddy currents, the heating coil is so wound that its turns decrease in diameter towards either or both of its ends, thereby minimising marginal dispersion or fringing of the magnetic field with respect to the charge. J. S. G. Thomas.

Electrolytic apparatus. F. Lawaczeck (U.S.P. 1,600,478, 21.9.26. Appl., 13.10.24. Conv., 20.10.23).—A number of cells, insulated from each other, are arranged in a pressure vessel containing electrolyte and communicate with the electrolyte therein. Current is supplied to the first of the series of cells and means are provided for collecting separately, under pressure, the gases liberated from the electrolyte. J. S. G. Thomas.

Electric furnace. J. K. Kiddle (E.P. 259,296, 6.7.25).—In a rotary electric furnace of the type in which the rotating element comprises a shell enclosing an electrical resistance and the refractory tube for containing the material to be treated, the rotating element has the form of a tubular shell reinforced by surrounding annular discs mounted thereon and braced together near the periphery by longitudinal stay-rods. J. S. G. Thomas.

Electrodeposition of chromium. C. L. Long, D. J. Macnaughtan, and G. E. Gardam (E.P. 258,724, 4.9.25).—Chromium is deposited electrolytically from an electrolyte having a chromic acid base to which is added a proportion not exceeding 3% by weight of copper, zinc, or nickel or any combination of these metals. Thus the electrolyte may consist of an aqueous solution containing 30% of chromic acid, 0·5—2% of chromium sulphate, and 0·25—2% of dissolved copper. The process permits the production of coherent deposits free from laminations and cracks by the use of high current densities, *e.g.*, 200 amp./sq. ft., at temperatures below 20°. J. S. G. Thomas.

Electrolytic separation of chromium. R. Appel (E.P. 259,118, 19.3.26. Conv., 26.11.25).—An adherent deposit of chromium is obtained by the electrolysis of a bath containing about 50 g. of chromic acid, or chromates, and 0·5 g./litre of sodium sulphate. Plates of rolled lead proved the most satisfactory material for anode material. The bath is maintained at 35—40° and the current density at 0·6 amp./sq. cm., and if at first a brown colloidal oxide be deposited, a few drops of sulphuric acid should be added to the bath. C. A. King.

Extraction of tin from alloys containing lead by electrolytic means. H. and S. Bondi and B. Neurath (E.P. 240,147, 31.8.25. Conv., 19.9.24).—An almost quantitative separation of tin and lead is effected if the electrolyte is such that the cathode potentials of the metals are made to differ so that the formation of a tin-lead alloy at the cathode is impossible. Thus, the addition of 10 g./litre of sulphuric acid, or 30 g./litre of sodium sulphate, or 30 g./litre of hydrogen iodide or potassium iodide to the hydrochloric acid electrolyte is effective. An extension of this process can be used in the separation of copper and antimony from tin by the addition of suitable substances capable of forming complex ions or salts with these metals. F. G. Crosse.

Electrolytic deposition of metallic coatings upon metals. A. C. Barlow (E.P. 259,307, 8.7.25).—The surface of the metal to be coated, *e.g.*, aluminium, magnesium, or an alloy of these metals, is roughened by the application of a weak solution of hydrofluoric acid, and the metal then used, without being washed or dried, as the cathode in an electrolytic bath containing boric acid as electrolyte and an anode of the metal to be deposited, *e.g.*, nickel, cobalt, or iron. Finally, the coated metal is washed in boiling water. J. S. G. Thomas.

Anodes for production of organic acids. F. Tallada (E.P. 257,826, 18.2.26).—The anode system consists of a conducting, insoluble core directly connected with the positive pole of a generator and an insoluble, perforated conducting casing or concentric series of such casings around, but out of direct contact with, the core, the space between core and casing being free or filled with an insoluble porous substance. The use of such a system prevents the formation of by-products in the production of organic acids, such as acetic, oxalic, or citric acids, by electrolysis of their salts. L. M. Clark.

Electrolyte for electrical batteries. G. A. HOWLAND (E.P. 259,099, 15.2.26).—An electrolyte for secondary batteries is described consisting of a mixture of soapstone, carbon dust, gypsum, sulphuric acid (d 1·125), snake oil, salicylic acid, sodium chloride, sodium chlorate, and potassium silicate solution.
J. S. G. THOMAS.

Depolarising agent for voltaic battery. T. A. EDISON, Assr. to T. A. EDISON, INC. (U.S.P. 1,599,121, 7.9.26. Appl., 26.2.24).—Chemically produced cupric hydroxide is combined with the hydroxide of an alkaline-earth metal. J. S. G. THOMAS.

Apparatus for electrical precipitation of suspended material from gases. C. H. WEISKOPF, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,600,496, 21.9.26. Appl., 28.11.23).—In an electrical precipitator a number of collecting electrode members have the form of parallel vertical plates with vertical flanges extending transversely thereto, and dividing the spaces between the plates into vertical flues. The flanges are not attached to the plates at their outer edges, in order to allow free vibration of the plates and flanges. J. S. G. THOMAS.

Electrolytic purification of graphite. L. C. HAFFNER (U.S.P. 1,600,730, 21.9.26. Appl., 31.7.22).—Impurities in a solution containing graphite in suspension are removed by electrolysis. J. S. G. THOMAS.

Cleaning and polishing silver, plate, etc. THE POLIVIT MANUF. CO., LTD. From F. J. M. VALENTIN (E.P. 258,726, 7.9.25).—Articles of silver, silver plate, etc. are cleaned electrochemically by immersion in an alkaline bath in the presence of aluminium and its alloys. Thus the articles may be immersed in a solution of washing soda to which, if desired, ammonia or an ammonium salt is added, in the presence of an alloy containing, *e.g.*, 95—97% Al, 3—2% Zn, 1—0·5% Cu, and 0·5—0·25% Ag. J. S. G. THOMAS.

Production of electrical insulating material. H. FRIEDLÄNDER, Assee. of A. JANSER (E.P. 236,224, 25.6.25. Conv., 26.6.24).—Fibrous material, *e.g.*, cardboard, felt, papier-mâché, etc., is impregnated with a hard wax or mixture of hard waxes, *e.g.*, montan wax, carnauba wax, together with, if desired, resins or their derivatives, metal salts of fatty acids, and derivatives of hard waxes, in the absence of alum and of substances liable to cause softening of the wax, so that the pores and voids in the fibrous material are completely filled.
J. S. G. THOMAS.

Manufacture of electrical insulators and other moulded articles. A. H. BROWN, and THE SILUMINITE INSULATOR CO., LTD. (E.P. 259,505, 8.7.25).—Portland or similar cement, together with, if desired, one or more non-binding fillers, *e.g.*, asbestos, powdered slate, clay, etc., is moulded and, after setting and hardening, is impregnated with one or more incompletely condensed liquid synthetic resins at a temperature of 60—80°, sufficiently high to cause the resin to permeate the whole, and finally heated to 100—130° to harden the resins.
J. S. G. THOMAS.

Arc welding. (E.P. 258,036 and 259,365 and U.S.P. 1,599,056).—See X.

XII.—FATS; OILS; WAXES.

Examination of ancient Egyptian (Tutankhamen) cosmetic. A. C. CHAPMAN and H. J. PLENDERLEITH (J.C.S., 1926, **10**, 2614—2619).—Examination of a 3300 years' old sterile cosmetic contained in a calcite jar showed the presence of (1) ether-soluble, saturated fatty matters (nearly 90%), (2) a resinous portion, (3) a small residue of calcium salts obviously derived from the action of free fatty acids present on the jar. The fatty matters consisted of (*a*) light petroleum soluble fatty acids, chiefly palmitic and stearic acids, (*b*) light petroleum insoluble "oxidised acids" apparently obtained by the oxidation of acids of the oleic type, and (*c*) glycerol, free and combined, to the extent of 5·46%. The very small amount of unsaponifiable matter present did not contain cholesterol or phytosterol. It is suggested that the cosmetic consisted of about 90% of a neutral animal fat with about 10% of some resin or balsam. H. BURTON.

[Detection of] whale oil. M. TSUJIMOTO (Chem. Umschau, 1926, **33**, 268—269).—In the determination of the iodine value of whale oil by the Wijs method a turbid solution results, whilst with vegetable oils the solution remains clear. This phenomenon now forms the basis of a method for detecting whale oil. The test is best applied to the freed fatty acids of the sample, the procedure being as follows: 0·5 g. of the acids from the oil is dissolved in 10 c.c. of ether and mixed with 3—5 c.c. of a glacial acetic acid solution of iodine monochloride. The mixture, at room temperature, is shaken at intervals during 2 hrs. In the presence of large amounts of whale oil the solution becomes turbid and deposits a fine precipitate. Smaller amounts give turbidity of longer duration, and finally a precipitate. With vegetable drying oils the solution remains quite clear if it is not kept for an excessive time, and if the temperature does not fall below 15°. A table is given showing the character of the precipitates from the fatty acids of linseed oil, Japanese sardine oil, herring oil, clupanodonic acid, and various mixtures, and it is demonstrated that the method detects 1% of sardine oil or 5% of herring oil in linseed oil, or 0·2% of clupanodonic acid in the acids of linseed oil. It is suggested that the actual yield of ether-insoluble iodochlorides is a useful value in fat analysis. These are white powders which become rose-pink when heated to 50—60°, and blacken without melting at 170—178°, giving off iodine vapour. The ether-insoluble iodochlorides from sardine oil and herring oil are essentially different from those of linolenic acid. E. HOLMES.

Rate of oxidation of linseed oil. W. ROGERS, JUN., and H. S. TAYLOR (J. Physical Chem., 1926, **30**, 1334—1347).—A simple apparatus (cf. Genthe, B., 1907, 56) giving true oxidation values for non-volatile oils is described, and the effect of various factors on the rate of oxidation has been studied. Increase in temperature increases oxidation and shortens, but does not eliminate, the induction period. As the concentration of the drier, in this case cobalt linoleate, is increased (i) the effect of increasing temperature becomes less marked, (ii) the induction period is shortened but never eliminated, and (iii) at a given temperature the rate of oxida-

tion approaches a maximum. This value is reached with 0·0003% of cobalt as linoleate, at 100°. Inhibitors (quinol, β-naphthol, and diphenylamine) are in general more effective at lower temperatures, and must be soluble in the oil to give reproducible results. There is evidence that the inhibitor does not act solely by the suppression of the catalyst, and both inhibitor and catalyst may work independently. With visible and ultra-violet light at the ordinary temperature, the rate of oxidation is not directly proportional to light intensity, and the reaction probably has a chain mechanism.

L. S. THEOBALD.

Reaction of the oils containing sulphur in olive oil. S. FACHINI (Giorn. Chim. Ind. Appl., 1926, 8, 428).—The oils containing sulphur are readily recognised by treatment of the oil in the presence of potassium hydroxide in alcoholic solution with lead, copper, or mercury, whereby the sulphur is converted into the characteristic sulphides. The reaction is of importance in modern methods of refining, for it is possible thereby to eliminate the last traces of sulphur. Other solvents, such as trichloroethylene and benzene, are suitable. The use of the characteristic chromate reaction as a means of distinguishing between oils extractable by solvents and those by pressure is confirmed (cf. B., 1926, 592).

P. V. McKIE.

Lime seed oil and oil cake. A. E. COLLENS (Analyst, 1926, **51**, 510—511).—A yield of about 70% of the oil present may readily be obtained from lime seeds (pips) by expression after preliminary heating. The filtered oil was brownish-yellow, and had the following characteristics :—$d^{27}_{15\cdot5}$ 0·9138; solidif. pt. —3°; n_D^{28} 1·4740; acid value 11·2; saponif. value 193·5; iodine value (Hübl) 109·7; solubility in alcohol 0·18%; unsaponif. matter 0·72%. The bitter taste of the oil may be removed by alcohol. Dominica press cake (30% expression of oil) contained nitrogen 4·1, potash 0·58, and phosphoric anhydride 0·89%, and analysis of a press residue shows moisture 15·08, ash 3·17, oil 14·2, crude protein 30·5, crude fibre 20·05, and carbohydrates 17%.

D. G. HEWER.

Saturated aliphatic alcohols from sperm whale oil and spermaceti. E. ANDRÉ and T. FRANÇOIS (Compt. rend., 1926, **183**, 663—665; cf. B., 1926, 247).—By esterification of sperm whale oil and spermaceti with acetic anhydride, fractional distillation, and hydrolysis, a small quantity of tetradecyl alcohol, m.p. 38° (*phenylurethane*, m.p. 70°), large quantities of hexadecyl alcohol, m.p. 49°, and a small quantity of octadecyl alcohol, m.p. 61° (*phenylurethane*, m.p. 79·5°), were obtained. No dodecyl alcohol was isolated. The *phenylurethane* of dodecyl alcohol has m.p. 78°

L. F. HEWITT.

Determination of mixtures of two and three [vegetable] oils by means of separation temperatures from various solvents. J. D. JANSEN and W. SCHUT (Chem. Weekblad, 1926, 23, 498—502).—Separation temperatures of mixtures of rape, arachis, cotton seed, sesamé, soya bean, and linseed oils with aniline (1 : 4) and with acetone (2 : 3) have been determined; temperature–composition curves for mixtures of two oils are given. For mixtures of three oils no suitable second solvent to be used in conjunction with aniline has been found, and the use of another constant, in conjunction with the temperature of separation from aniline, is suggested.

S. I. LEVY.

Petroleum hydrocarbons from fats. MARCUSSON and BAUERSCHÄFER.—See II.

Deliquescent properties of magnesium chloride, calcium chloride, and glycerol. SWAN.—See VII.

PATENTS.

De-acidification of oils and fats. H. BOLLMANN (E.P. 258,786, 3.3.26).—The losses due to formation of emulsions and to splitting when oils and fats are de-acidified by the action of alkalis under heat are avoided by using as de-acidifier a freely flowing aqueous composition containing an acid fixer, *e.g.*, sodium hydroxide solution, intimately mixed with a heavy material such as talc, bole, powdered marble, metal powder, etc., in the presence or absence of mucilaginous matter, *e.g.*, gelatin, agar-agar, etc. With slight warming, the soaps arising from the free acid originally present are brought down by the heavy material, and may be readily removed by filtration.

S. S. WOOLF.

Oxidation of fats, oils, waxes, resins, etc. J. Y. JOHNSON. From BADISCHE ANILIN & SODA FABRIK (E.P. 258,099, 16.10.25).—A gas containing oxygen is blown through the heated mixture of oils, waxes, resins, etc. to be oxidised, in which are placed a number of disconnected rings, balls, etc. of clay, glass, earthenware, or metal (particularly aluminium or its alloys). The heat developed in the reaction is utilised for preheating a new charge.

S. S. WOOLF.

Deodorising, clarifying, and neutralising animal and vegetable oils, fats, and waxes. E. FORAY (F.P. 601,919, 4.8.25).—The oils or liquefied fats are mixed with animal or vegetable charcoal, with or without the addition of a catalyst such as manganous oxide, and separated by filtration.

A. RAYNER.

Treatment of oily vegetable materials for the separate recovery of their constituents. L. F. DAVID and G. FÉLIZAT (F.P. 603,836, 29.12.24).—The materials are ground up with alkaline solution, by which the oils are emulsified and the albumins dissolved. The cellulose is recovered by sieving, the starch by decantation, the oil by centrifuging, and the albumins by precipitation by an acid.

A. RAYNER.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Chemistry of satin white. P. FUCHS (Chem.-Ztg., 1926, **50**, 769—770).—The formation of satin white from aluminium sulphate and milk of lime takes place only when the sulphate is basic and contains 80—85% of the sulphuric acid content of the normal salt. Determination of the calcium sulphate in the final product shows that the reaction is: $Al_2(OH)_2(SO_4)_2,Al_2(SO_4)_3 + 11Ca(OH)_2 = 5(CaSO_4,2H_2O) + 2Al_2Ca_3O_6 + 2H_2O$ (cf. Cobenzl, B., 1920, 664). Satin white is a mixture of gypsum and calcium aluminate in the molecular ratio 5 : 2, together with small varying amounts of calcium hydroxide.

F. R. ENNOS.

Coniferyl reaction of some resins. F. REINITZER (Z. anal. Chem., 1926, **69**, 114—121).—The following

gums and resins give a red colour with phloroglucinol and hydrochloric acid (coniferyl reaction): Siam and Sumatra benzoin, tolu and Peru balsams, storax, asafetida, and galbanum, the resins extruded from pines, firs, and larches, acaroid resin, dammar, Manila elemi, copaiba balsam, and olive resin. A weaker reaction is given by olibanum, myrrh, and mastic. The reaction is due to the presence of coniferyl alcohol or a substance bearing a close relationship to it in the case of all the resins in the above list down to and including acaroid. The reaction of elemi is due to the presence of elemicin and of myrrh to the presence of eugenol; the cause of the reaction in the other resins could not be ascertained. Ferulic acid and *p*-cumic acid do not give the reaction, and it is doubtful whether pure caffeic acid does, although the crude acid usually gives a feeble red colour.

A. R. POWELL.

Rate of oxidation of linseed oil. ROGERS.—See XII.

PATENTS.

Anti-corrosive preparations. S. FOWLER and E. EDSER (E.P. 258,795, 24.3.25).—Mixtures of neutral soap-free wool fat (adeps lanæ) with non-volatile thinning agents, *e.g.*, neutral lubricating oil, afford a protective layer to metal surfaces. A preferred method of purifying the wool fat is that described in E.P. 253,995 (B., 1926, 759).

S. S. WOOLF.

Protective coating or impregnating compositions. A. C. and M. HOLZAPFEL (E.P. 259,292, 6.7.25).—An alkali fluoride is dissolved in oleic acid, and made up as a paint with varnish gum and a usual solvent. As a wood preservative the fluoride solution is mixed with any impregnating oil suitable for the purpose, zinc oxide being sometimes added to give the required viscosity.

F. G. CROSSE.

Preparation of lacquers from tung oil. W. SCHMIDDING (E.P. 247,599, 13.2.26. Conv., 13.2.25).—Tung oil is rapidly heated almost to gelatinisation, and when the desired consistency is reached the other molten or heated raw materials, such as resins, resinates, oils, etc., are run in from a secondary varnish kettle situated above the kettle proper.

S. S. WOOLF.

Manufacture of covering materials. KÖLN-ROTTWEIL A.-G. (E.P. 241,528, 1.9.25. Conv., 17.10.24).—Oxidised oils, with or without the addition of a resin, are mixed with cellulose nitrate, plasticisers, and, if desired, filling and colouring materials, and the mass, after being rendered homogeneous on hot rollers, is finely ground and moulded for use as a resilient covering material.

S. S. WOOLF.

High-grade wood rosin. HERCULES POWDER CO., Assees of H. E. KAISER and R. S. HANCOCK (E.P. 253,082, 19.4.26. Conv., 5.6.25).—Low-grade wood rosin containing objectionable colouring matter is dissolved in gasoline, and furfuraldehyde is added. On refrigeration in one or more stages, some of the resin and a major portion of the furfuraldehyde, holding in solution all the colouring matter, settle out. On removal of this layer, high-grade wood rosin suitable for use in high-grade soap manufacture may be recovered from the gasoline solution to the extent of 70—75% of the origiual rosin. Medium-grade rosin may be obtained from the precipitate by gasoline extraction. Both solvents are recovered.

S. S. WOOLF.

Resinous compositions. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of C. F. PETERSON (E.P. 250,949, 14.4.26. Conv., 14.4.25).—Relatively small proportions of phenol–formaldyhyde resin are added to glycerol–phthalic anhydride resins. The polymerisation ("curing") of the latter is accelerated, and the resulting material has less tendency to soften under heat than the usual cured "glyptal."

S. S. WOOLF.

Producing resinous substances.—CHEM. FABR DR. K. ALBERT, G.M.B.H., A. AMANN, and E. FONROBERT (E.P. 259,030, 5.10.25).—The still acid reaction product of rosin (colophony), or a similar natural acid resin, and resins of the phenol–carbonyl type is neutralised by a reagent each molecule of which is capable of combining with at least two resin acid groups, *e.g.*, polyhydric alcohols or oxides of polyvalent metals, glycerol and zinc oxide being typical. Before the final neutralisation. the mass may be distilled *in vacuo* to remove all or part of the volatile constituents of the natural resins which have not been combined with the phenol–carbonyl compound. The mol. wt. of the resulting resin is greater than that of molten natural copals.

S. S. WOOLF.

Manufacture of articles from synthetic resins. BRIT. CYANIDES CO., LTD., and E. C. ROSSITER (E.P. 258,950, 1.7.25).—The conversion of the first (water-soluble, fusible) or second (water-insoluble, fusible) stage condensation product of thiourea and formaldehyde into the final (insoluble, infusible) stage is assisted by the incorporation with the resin-intermediate and filling materials of an acid condensing agent such as oxalic acid, sodium hydrogen sulphate, potassium tetroxalate, aniline hydrochloride, or similar compounds.

S. S. WOOLF.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Influence of diphenylguanidine on the mechanical properties of vulcanised rubber. L. STOLL (Gummi-Ztg., 1926, 41, 193—196).—Investigation is made of the effect of variations in temperature of vulcanisation and in the proportion of diphenylguanidine and the accompanying sulphur and zinc oxide on the tensile strength, extensibility, and other physical characteristics of the vulcanised product. The first small proportions of zinc oxide have a much more marked effect then subsequent increments. Reversion on vulcanisation with small proportions of sulphur is less marked with diphenylguanidine than with "vulcone." An increase from 55 to 65 lb./sq. in. in the pressure of the steam used for vulcanisation results in vulcanisation proceeding 1·8 times as rapidly. The optimum proportion of diphenylguanidine is 1·5—2% (on the rubber), and of sulphur approximately 5%.

D. F. TWISS.

PATENTS.

Treatment of rubber and apparatus therefor. DUNLOP RUBBER CO., LTD., H. C. YOUNG, and J. D. CAMPBELL (E.P. 259,028, 3.10.25).—Rubber is mixed with all the intended ingredients, excepting sulphur, by means of a machine of the ordinary or the "internal mixer" type; the sheeted mixing then passes along

a cooling conveyor to another mill, or by a circuitous route back to the sheeting mill, where the sulphur can be introduced without fear of premature vulcanisation. D. F. Twiss.

Treatment of rubber and the like and apparatus therefor. Dunlop Rubber Co., Ltd., and A. E. Penfold (E.P. 259,022, 26.9.25).—In calendering rubber into sheet form the material is batched up inside a roll of damp cloth instead of in the usual roll of "faced" sheet. D. F. Twiss.

Vulcanisation of rubber. R. V. H. Heuser, Assr. to A. C. Burrage (U.S.P. 1,597,233, 24.8.26. Appl., 27.2.23).—*s*-Phenyl-*o*-tolylguanidine is used as vulcanisation accelerator.

XV.—LEATHER; GLUE.

Chemical nature of vegetable tanning. A. W Thomas (J. Amer. Leather Chem. Assoc., 1926, **21**, 487—516).—The rate of fixation of commercial tannins by pelt in a given time has been measured at different p_H values and shows a minimum at p_H 5·0 rising to a maximum at p_H 2·0. From the Procter–Wilson theory it follows that pelt will have a gradually increasing potential difference as the p_H decreases to 2·0, consequently the reaction of the positively charged collagen with the negatively charged tannin particles should exhibit a minimum rate of fixation at p_H 5·0 and increase from 5·0 to 2·0. This explains the rate of tanning at p_H values$<$5·0, but not in the alkaline region. The increase in the rate of fixation of commercial tannins from p_H 5·0 to 8·0 is attributed to the β form of collagen suggested by Wilson and Gallun (cf. B., 1923, 236 A), and having its isoelectric point at p_H 7·7. With increasing amounts of β, positively charged, in the solution, as the p_H increases from 5·0 to 7·7, reaction will take place at an increasing rate with the negatively charged tanning particles. Experiments with quinone-free tannins, *e.g.*, gallotannic acid, show no fixation of tannin on the alkaline side of p_H 8·0, but commercial tannins contain quinonic compounds, and it is probably these which combine with the pelt at values of $p_H$$>$8·0. All these facts favour a chemical theory of tanning in which there is chemical combination between the amino- and possibly imino-groups of the complex collagen cations, and the anions of the complex weak organic acids, the tannins, the rate of combination being a function of the p_H value of the tannin solution. Experiments with de-aminised calf skin show that the isoelectric points are p_H 4·0 and 8·3, and the point of miniumum fixation of tannin is p_H 3·5, the amounts of tannin fixed in a given time are consistently less than with ordinary calf skin, and the rate of fixation of tannin increases rapidly on the alkaline side of p_H 3·5, rising to a maximum at p_H 8·0 and dropping sharply at p_H 9·0 and 10·0. These results show the importance of the amino-groups in the tanning process. De-aminised collagen combines with less quinone than untreated collagen, thus confirming Meunier's theory of the quinone tannage. D. Woodroffe.

Physical and chemical properties of vegetable tanned insole bellies. V. Wear-resistance. D. Woodroffe (J. Soc. Leather Trades Chem., 1926, **10**, 266—272).—The wear-resistance of various samples of vegetable-tanned insole bellies was determined by holding the leather on a rapidly revolving buffing wheel fitted with a fine carborundum paper. The paper was changed every minute to avoid clogging. The time was noted for the leather to wear through and the time calculated for one-inch thickness to be worn through. No relationship could be traced between the wear-resistance and the degree of tannage, contrary to Powarnin and Schichireff's claims (cf. B., 1926, 683). The wear-resistance increases as the content of water-soluble matter increases, attains a maximum with leathers containing about 20·5% of water-soluble matter, and then diminishes with higher water-soluble content. D. Woodroffe.

Extraction of sumac for analysis. Comparison of various methods. T. G. Parker and L. Winch (J. Soc. Leather Trades Chem., 1926, **10**, 272—280).—Comparative analyses of different samples of sumac have been made, using infusions obtained by Procter's method, a continuous percolation method, and the present official method of intermittent siphoning. The last-named method extracted more soluble matter, but gave a slightly lower tannin content than the Procter method or the continuous method. Extraction of sumac at 45° did not remove all the tannin. Soaking overnight was necessary to obtain concordant results. The period of soaking should not exceed 16 hrs., and the temperature of the water should not exceed 13°. The minimum time for the extraction of 14—16 g. of sumac is 3 hrs. The method of extraction prescribed by the American Leather Chemists' Association gave a higher yield, both of tannin and extracted matter, and it possesses several advantages. A more thorough extraction is obtained by using 2 litres of water. D. Woodroffe.

Treatment of tannery waste. Mohlman.—See XXIII.

Patents.

Treatment of bones and the like for the production of bone meals and the like. W. H. Boorne (E.P. 258,686, 1.7.25).—Ground bone meal is degreased with boiling water, and chlorinated water is introduced into the pans. Where steam is used for degreasing, chlorinated water or chlorine gas may be introduced simultaneously, but if solvents such as benzine are used the meal is first degreased, then chlorinated. D. Woodroffe.

Production of aqueous solutions or dispersions of isinglass. W. M. Carpenter (E.P. 258,444, 17.11.25).—A special apparatus is used comprising two vessels arranged one above the other, each provided with vertical shafts coaxially located, the upper shaft being furnished with a propeller, and the lower one is fitted with devices adapted to travel over the surface of a perforated partition in the base of the lower vessel. A driving connexion can be established or interrupted between the shafts. The isinglass is agitated in the upper vessel with a mixture of water and acid until sufficiently swollen (six days), then delivered into the lower vessel. The partition in the lower vessel forms a cone or hopper through which the swollen isinglass is discharged. D. Woodroffe.

Transforming glue, gelatin, etc. in melted or solution form to granules or pearls. Akt.-Ges. f. Chem. Produkte (E.P. 244,406, 29.6.25. Conv., 15.12.24).—Glue, gelatin, etc., melted or in solution form, is dropped from a device comprising a series of nozzles, arranged vertically above a revolving cooling drum mounted on a horizontal axis, or a belt conveyer or rotary table which is not coated with any solid or liquid lubricant. The cooling surface can be arranged to pass through a cooling bath. The resulting granules are detached from the cooling surface when sufficiently solidified. D. Woodroffe.

Manufacture of glue or gelatin in the form of discs or tablets. Comp. Nat. Mat. Col. et Manuf. de Prod. Chim. du Nord Réunies, Etab. Kuhlmann (E.P. 239,531, 4.9.25. Conv., 4.9.24).—A solution of glue or gelatin is fed, drop by drop, on to a suitable surface, *e.g.*, a cylinder or metallic or canvas belt, cooled below 15°, and coated with some oily or waxy substance. The drops solidify to form small tablets, which are detached and dried. D. F. Twiss.

XVI.—AGRICULTURE.

Infertility of the subsoil. C. R. Van der Merwe (J. Agric. Sci., 1926, **16**, 507—521).—The author has compared the physical and chemical properties of the soil and subsoil of a ferruginous clay soil occurring in the Transvaal. The data relate to virgin soil. The infertility of the subsoil is not considered to be due to the presence of " active " aluminium compounds, or to deficiency in potash. Chemical analyses and pot experiments show that the principal cause of infertility is deficiency in available phosphate. Available nitrogen is also deficient owing to bacterial inactivity, but no beneficial effect is produced by the use of nitrogenous dressings in the absence of adequate dressings of phosphate. G. W. Robinson.

Scottish drift soil. IV. Exchangeable bases. J. Hendrick and G. Newlands (J. Agric. Sci., 1926, **16**, 584—595).—Data are given for the exchangeable base content of a Scottish drift soil. The presence of silicon, aluminium, iron, and manganese is noted in the extracts in addition to calcium, magnesium, potassium, and sodium. The relative proportions of the different exchangeable bases are: calcium, 85·02%; magnesium, 8·11%; potassium, 2·18%; sodium, 4·68%. The results are in agreement with those generally found for acid soils. G. W. Robinson.

Soil " humus." III. Nature of the substances contributing to formation of humus. S. A. Waksman (Soil Sci., 1926, **22**, 323—333; cf. B., 1926, 892).—The decomposition of various straw preparations by pure cultures of some soil organisms, including both fungi and bacteria, has been investigated. Rye straw was subjected to a series of extractions, and the residue after each treatment used for the study of its decomposition by the organisms in sand or soil media. The lignins are the most resistant of the constituents of straw; their accumulation accounts for much of the soil " humus " formed as a result of the decomposition of the straw. Moreover, the decomposition of celluloses and hemicelluloses by fungi in the soil is to some extent prevented in the presence of lignins. The breaking down of the celluloses and other constituents of straw involves the conversion of considerable amounts of nitrogen into microbial protoplasm, which also forms part of the soil " humus." C. T. Gimingham.

Availability of nitrogen in green manures. F. Löhnis (Soil Sci., 1926, **22**, 253—290).—The results are recorded of many series of vegetation experiments on the effect on the yield and composition of a number of different crops, of incorporating various leguminous plants with the soil as green manures. A poor heavy clay and a fertile greenhouse soil were used. The experiments extended over ten years. The legumes were grown on field plots, harvested at three stages of growth, and dried before use; they were applied at rates equivalent to 4½, 1½, and ½ tons of air-dry material per acre. The availability of the nitrogen of the green manures showed wide variations dependent on the quality and quantity of the material and on the character of the soil. An average return of 50—60% of the nitrogen applied was recorded, but not more than 50—75% of this amount was contained in the first crop. Crop increases were larger and more lasting on the poor soil. In a soil not too poor in humus, green manures tend to accelerate the activities of the micro-organisms, with the result that there is an intensified nitrification of the humus nitrogen, and more nitrogen may be found in the first crop increases than has become available from the green manures. C. T. Gimingham.

Lime–sulphur–calcium arsenate spray. W. Goodwin and H. Martin (J. Agric. Sci., 1926, **16**, 596—606).—It is improbable that stable tricalcium arsenate can be formed by precipitation from aqueous solution. Calcium hydroxide and dicalcium arsenate give a series of basic arsenates which are hydrolysed in aqueous suspension. Addition of lime to dicalcium arsenate thus reduces, temporarily, the amount of arsenic in solution, but on exposure to atmospheric carbon dioxide, the original solubility is restored. Addition of " lime sulphur " to lime–calcium arsenate spray reduces the concentration of soluble arsenic, and hence the risk of injury to foliage. The precipitation of sulphur from calcium polysulphides is unaffected by calcium arsenate. The reduction in the precipitation of sulphur by lime is dependent on the rate of carbonation of the lime on the leaf surface. The fungicidal action of calcium polysulphides is thus unaffected by calcium arsenate, but may be reduced by an excess of lime. G. W. Robinson.

Losses in the tower silo. H. E. Woodman and A. Amos (J. Agric. Sci., 1926, **16**, 539—550).—The destruction of carbohydrate by oxidation during ensilage bears no relationship to the temperature attained during the process. " Acid brown " silage and " sweet " silage can be made with an average loss of 5—6% of the dry matter originally present. With " green fruity " silage the average loss is 8—9%. Losses of dry matter may be excessive if very wet material is preserved. The optimum dry matter content is about 26—34%. A period of about a fortnight is apparently sufficient for a green crop to acquire the character of silage. G. W. Robinson.

Seasonal changes in the composition of winter wheat plants in relation to frost resistance. R. Newton and W. R. Brown (J. Agric. Sci., 1926, 16, 522—538; cf. B., 1924, 484).—The authors have studied the seasonal changes in the distribution of the more important constituents between physiologically active cell fluids and supporting tissue in wheat plants. One of the most important changes is the reduction in moisture content, which is greatest in hardy varieties, and, by causing a concentration of colloids and sugars in cell fluids, increases resistance to frost. The cell colloids are mainly proteins, 90% of the total protein of the plants being present in the fluids. Pentosans are mainly in structural tissue, although pentose sugars occur in small concentrations in the fluids. Ash is mainly in the fluids. The accumulation of sugar in the autumn is greatest in hardy varieties, and tends to protect proteins against denaturation by frost. No connexion was observed between lipoid or amino-and coagulable-nitrogen changes and frost resistance. G. W. Robinson.

Non-volatile constituents of the cotton plant. F. B. Power and V. K. Chesnut (J. Amer. Chem. Soc., 1926, 48, 2721—2737).—The raw material for this investigation consisted of the foliage, "squares," and flowers of the cotton plant, all the bolls which had been developed having been removed. The loss in weight on drying was nearly 80%. Preliminary tests indicated the presence of chlorine and the absence of enzymes capable of hydrolysing β-glucosides. The main bulk of the dried material (29 kg.) was extracted by percolation with 95% alcohol. This extract was acid, but the distillate therefrom was alkaline owing to the presence of ammonia and trimethylamine (cf. Power and Chesnut, B., 1925, 612). Steam distillation of the concentrated alcoholic extract afforded an essential oil (8·9 g.) together with formic and acetic acids, whilst the residue consisted of an aqueous solution (A) and a black, oily resin (B). (A) contained potassium nitrate and potassium chloride in considerable amount, quercetin, betaine, choline, and succinic acid, together with a trace of salicylic acid and of an *acid*, m.p. 105—106°, and a neutral, crystalline substance, m.p. 140—143°. Reducing sugars were also present. (B), amounting to 3·134% of the dried material, contained a *phytosterol*, $C_{27}H_{46}O,H_2O$, m.p. 135°, $[\alpha]_D^{20}$ − 32·9° in chloroform (*acetyl* derivative, m.p. 121°), a phytosterol glucoside, m.p. 218—223°, apparently identical with "ipurganol" (cf. Power and Rogerson, B., 1910, 232), pentatriacontane, $C_{35}H_{72}$, palmitic acid, a *phenolic acid*, m.p. 188—189°, butyric, valeric, and *n*-hexoic acids, and a phenolic *substance*, not identical with that previously isolated (Power and Chesnut, *loc. cit.*). Vacuum distillation of a portion of the unsaponifiable material afforded dipentene, a *substance*, $C_8H_{10}O$, b.p. 200—220°/5—9 mm., d_{15}^{15} 0·9153, a *sesquiterpene*, $C_{15}H_{24}$, b.p. 250—280°/5—9 mm., d_{15}^{15} 0·8909, n_D^{20} 1·4891, $[\alpha]_D^{20}$ + 0·28°, and *diterpenes*, b.p. 300—350°/5—9 mm., d_{15}^{15} 0·9120. F. G. Willson.

Action of citric acid solutions of varying ammonia content on di- and tri-calcium phosphate. A. H. M. Andreasen (Z. anal. Chem., 1926, 69, 107—113).—The solubilities of tricalcium phosphate and of dicalcium hydrogen phosphate in mono-, di-, and tri-ammonium citrate and in citric acid have been determined by shaking the precipitated phosphates with citrate solutions of varying concentration for periods of 0·5, 4, and 20 hrs. In all cases solution is shown to be due to the conversion of the calcium phosphates into monocalcium tetrahydrogen phosphate with the simultaneous formation of acid calcium citrates which, in the more concentrated solutions, slowly crystallise out in the form of needles. There is also some evidence of the formation of complexes containing 1 mol. of tricalcium phosphate and 4 mols. of citric acid or 1 mol. of dicalcium phosphate and 1 mol. of citric acid, but no solid compounds of these compositions could be isolated. A. R. Powell.

Argillaceous colloids of the soil. Demolon (Chim. et Ind., 1926, 16, 552—555).

Decomposition of cellulose in soil. Winogradsky. —See V.

Patents.

Transforming organic matter [to render the nitrogen available for fertilising]. E. P. Cooke, Assr. to American Beccari Corp. (U.S.P. 1,597,724—5, 31.8.26. Appl., 2 and 13.6.23).—A fermentation process for transforming organic matter into a form in which its nitrogen is available for use as a fertiliser consists in supplying air to the material so as to favour the growth of the micro-organisms concerned, and in regulating the temperature in accordance with their requirements (*e.g.*, at about 63°). C. T. Gimingham.

Breaking up cellulosic constituents in vegetable substance for production of manure. Fäkaltorf-Studienges.m.b.H., and A. Baumgarten-Crusius (E.P. 258,149, 21.1.26).—The process consists in propagating aerobic and anaerobic types of cellulose-fermenting bacteria from a variety of sources, combining them into a mixed culture, and adding the culture to the disintegrated vegetable material which it is desired to decompose. C. T. Gimingham.

Fertiliser. G. Barsky, Assr. to Amer. Cyanamid Co. (U.S.P. 1,599,198, 7.9.26. Appl., 28.7.23).—In order to eliminate the risk of fire in the manufacture of a fertiliser containing a nitrate, calcium cyanamide is added to the material to the extent of 50%. F. G. Crosse.

Fertiliser. J. M. Braham and F. E. Allison (U.S.P. 1,598,638, 7.9.26. Appl., 6.12.23).—A mixture of cyanamide and calcined phosphate obtained by calcining a mixture of phosphate rock, together with an alkali metal salt and carbonaceous matter, is suggested for the composition of a fertiliser. F. G. Crosse.

Manufacture of phosphatic fertilisers. O. R. Olsen and E. Torkildsen (E.P. 258,144, 11.1.26).—Ground phosphate rock is mixed with at least 25% of its weight of an alkaline-earth carbonate and rapidly heated in an oxidising atmosphere to a temperature between 1000° and 1600°. Addition of an oxide of iron reduces the temperature of the reaction. The product contains a high proportion of citric acid-soluble phosphoric acid. C. T. Gimingham.

XVII.—SUGARS; STARCHES; GUMS.

Polarimetric determination of sucrose in sweetened condensed milk. P. Honegger (Analyst, 1926, **51**, 496—503).—The method used is a modification of that of Revis and Payne (cf. B., 1914, 1166), whereby 26 g. of condensed milk are made up to 90 c.c. with boiling water and heated for 3 min. 3 c.c. of acid mercuric nitrate are added to the cooled solution, the volume made up to 100 c.c. at 20°, and 4 c.c. of water at 20° added. After shaking vigorously for 1 min. and filtering the polarimetric reading is made at 20°. After inversion (7 min. in boiling water), cooling, and filtering, the reading is again taken. Results obtained by this method agreed closely with those by the gravimetric method except from March to September, and the irregularity was probably due to variability of the volume of the condensed milk derived from the original fresh milk, the fat and colloidal substances being the chief factors. To overcome this the method may be modified so that quantities are made up to definite weights after dilution.
D. G. Hewer.

Absorption of water by dried films of boiled starch. II. Absorption and desorption between 20° and 90°. E. Swan (J. Text. Inst., 1926, **17**, 527—536 T).—The absorption curves (connecting moisture taken up with atmospheric humidity) for starch films are plotted for the range 20—90°. They are S-shaped, and indicate that except at humidities above 90% R.H. starch becomes less hygroscopic as the temperature is raised. At about 90% R.H. there is some evidence of the curves for higher temperatures crossing those for lower. A new set of curves (desorption) is obtained on bringing the starch back from moist conditions to dry. These lie higher than the absorption curves (regains being the ordinates). The hysteresis depends on the treatment of the film. If this has had prolonged heating or been evaporated at a high temperature it absorbs less on being placed in a moist atmosphere, but retains more on "drying" again. This effect of heat treatment in lowering the hygroscopicity of starch or increasing its retentive power may be expected to have practical significance in the sizing and starching of cotton goods.
J. C. Withers.

Occurrence of gentiobiose in the products of the commercial hydrolysis of corn [maize] starch. H. Berlin (J. Amer. Chem. Soc., 1926, **48**, 2627—2630).—The unfermentable portion of "hydrol"—the final mother-liquor in the manufacture of dextrose from maize starch—contains about 18·3% of gentiobiose, which was identified as the octa-acetate. When treated with phenylhydrazine, the purified, unfermentable residue from "hydrol" yields a *phenylosazone*, $C_{24}H_{32}N_4O_9,H_2O$, m.p. 150—152°, $[\alpha]_D^{25}$ −20·24° in methyl alcohol, closely resembling Fischer's *iso*maltosephenylosazone (A., 1891, 412). "*iso*Maltose" thus appears to be a mixture of carbohydrates.
F. G. Willson.

Patents.

Crystallisation of sugar solutions etc. F. Lafeuille (E.P. 245,114, 18.12.25. Conv., 24.12.24).—Apparatus for crystallising sugar massecuites or other supersaturated solutions comprises a rotary horizontal cylinder provided with means for producing a uniform cooling both from one transverse section to any other transverse section and from the periphery to the centre of the same section, cooling being maintained during the entire operation of the apparatus. Cooling is effected by arranging the tubes in such a way that on any circle concentric with the apparatus, traced on any section, there are two tubes which are symmetrical relative to the axis of the apparatus, cooling water circulating through these in opposite direction. Alternatively, the tubes are arranged in spirals so as to admit the cooling water simultaneously through the outermost tube of one of the sets of tubes and through that tube of the other set which is nearest the centre end of the apparatus. Three other modifications of the tubular cooling system are described.
J. P. Ogilvie.

XVIII.—FERMENTATION INDUSTRIES.

Fermentation of cellulose and its derivatives. H. B. Speakman (Canad. Chem. Met., 1926, **10**, 229—231).—See B., 1926, 687.

Patents.

Recovery of alcohol, organic acids, and fertiliser from fermented saccharine materials. G. T. Reich (U.S.P. 1,599,185, 7.9.26. Appl., 15.6.25).—The neutralised fermentation liquid is evaporated and a fraction collected containing the bulk of the alcoholic content. The organic acids and fertilising materials are recovered from the calcined evaporation residue.
C. Ranken.

Dealcoholising beverages (U.S.P. 1,598,601).—See XIX.

XIX.—FOODS.

Nutrition studies and statistics. I. Chemical composition of wheat grains. II. Grain size and weight per hectolitre of wheat. III. Cellulose content of wheat grains. L. Berczeller and H. Wastl (Biochem. Z., 1926, **177**, 168—180, 181—189, 190—195).—I. From a study of Zaharia's figures ("Le Blé roumain," Bucharest, 1910) it is shown that the average chemical composition of wheat of one season, or of a number of seasons, shows characteristic deviations from the binomial curve with regard to protein, cellulose, fat, and ash content. The extent of the variations, which differs for individual components, is least in the case of fat and greatest in the case of protein. Only in the former case was a relationship found between the extent of the variations and the maximal frequency value. II. The binomial curve does not apply to grain size nor to weight per hectolitre, nor do the values found show the same regularities as the chemical properties. III. Zaharia's figures show a marked increase in maximal frequency value of the cellulose content of wheat grains over the period 1900—1905.
J. Pryde.

Milling and panification with cereals other than wheat. D. Marotta (Annali Chim. Appl., 1926, **16**, 333—399).—Results are given of a large number of milling and baking experiments with mixtures of different cereals, with a view of economising wheat. The best mixtures for baking purposes are those of wheat with either rye or barley or both, the mixture containing at least 50% of wheat flour. Satisfactory results are still obtained if one-half of the rye or barley in such mixtures

is replaced by maize or rice. Numerous photographs and analytical results are given. T. H. Pope.

Breadmaking. d'Arbouet (Rev. gén. Colloid., 1926, 4, 199—201, 225—228).—The principles underlying the making of bread are discussed. Special importance is attached to the work of Lüers and Schwartz (B., 1925, 469), and to the investigations of Dearsley on the value of p_H determinations as indicative of the baking values of flours. A. G. Pollard.

Application of the Feder ratio to tinned sausages. A. Gronover and E. Wohnlich (Z. Unters. Lebensm., 1926, 52, 53—60).—The validity of the use of the normal Feder ratio values for cooked meats in the examination of tinned sausages is discussed. During the sterilisation of sausages in tins considerable absorption of water takes place, often increasing the moisture content by as much as 20%. The rate and extent of absorption of water are irregular and vary with the method of preparation and treatment of the meat, but appear to be less where the fat content of the meat is high. A. G. Pollard.

Solubility of the copper in basic copper carbonate [used for treating wheat]. T. D. Hall (J. S. Afr. Chem. Inst., 1926, 9, 10—17).—Wheat previously dusted with basic copper carbonate for the control of covered or stinking smut, *Tilletia tritici*, and germinated in paper for 9—11 days at 22—26·5°, gives a water-soluble extract containing copper, the amount of which is increased if germination is carried out in moist soil. Results, expressed in mg. of copper, show that approximately 5—12% of the copper attached to the wheat grains during dusting is made soluble on germination. H. Burton.

Colorimetric determination of iron [in tea etc.] by the ferrocyanide method. W. R. Mummery (Analyst, 1926, 51, 511—512).—Walker's modification of the ferrocyanide method (B., 1925, 573) makes use of nitric acid for the extraction of the ash. This was found to cause low results; a mixture of 5 c.c. of concentrated hydrochloric acid and 2 c.c. of distilled water (sufficient for two extractions of the ash) should be used, followed by evaporation to dryness and subsequent solution in 3 c.c. of concentrated nitric acid. The ash of tea was the only ash which was found to yield its total iron on extraction with nitric acid. D. G. Hewer.

Nutrients required for milk production with Indian foodstuffs. E. J. Warth, L. Singh, and S. M. Husein (Mem. Dept. Agric. India [Chem.], 1926, 8, 153—187).

Determination of mixtures of two and three [vegetable] oils by means of separation temperatures from various solvents. Jansen and Schut.—See XII.

Sugar in condensed milk. Honegger.—See XVII.

Chemical characteristics of some trade wastes. Buswell, Greenfield, and Shive.—See XXIII.

Treatment of packing-house and corn-products waste. Mohlman.—See XXIII.

Disposal of some organic trade wastes. Bartow.—See XXIII.

Patents.

Food product. W. D. Richardson, Assr. to Swift & Co. (U.S.P. 1,599,030, 7.9.26. Appl., 4.8.25).—A mixture of blood and carbohydrate material is fermented by means of lactic acid bacteria, the fermentation being allowed to proceed to completion and the product dried and ground. C. Ranken.

Food product. K. K. Jones, Assr. to Swift & Co. (U.S.P. 1,599,031, 7.9.26. Appl., 4.8.25).—A mixture of blood and carbohydrate material is fermented by means of yeast, and the resulting product dried. C. Ranken.

Treatment of fruit for the prevention of decay. W. R. Barger and L. A. Hawkins, Assrs. to People of the U.S. (U.S.P. 1,598,697, 7.9.26. Appl., 25.1.26).—The decay of fruits by green mold caused by *Penicillium digitatum*, Sacc., is prevented by treating them with a solution of 1 part of borax and 2 parts of boric acid in water, such solution containing 8% of the combined substances. F. G. Crosse.

Dealcoholisation of beverages and the like. C. H. Caspar (U.S.P. 1,598,601, 7.9.26. Appl., 27.4.20).—The fermenting liquid is circulated through the gases and vapours generated by fermentation and the alcohol condensed therefrom. The temperature of the liquid is maintained at a point less than that at which ebullition occurs. C. Ranken.

Purification of phosphatides [from soya beans etc.]. H. Bollmann (E.P. 259,166, 9.8.26. Conv., 6.10.25).—Phosphatides, accompanied by water, oil, free fatty acids, bitter substances, etc., are separated from the main quantity of an oil, and subjected to distillation under reduced pressure. Water may be added to the phosphatide-containing mixture prior to the distillation, or during it. The distillate may be treated with strong alcohol under heat, the undissolved matter separated, the solutions fractionally cooled, and the products precipitating out during cooling later separated, animal charcoal or other decolorising agent being added before such cooling takes place. J. P. Ogilvie.

Nitrogenous extracts from albuminoid substances. Soc. Franç. des Prod. Alimentaires Azotés (E.P. 246,779, 15.4.25. Conv., 29.1.25).—A solid albuminous substance, *e.g.*, fish, is boiled with water; the fatty matters liberated are removed and the residue is then subjected to heterolysis by proteolytic ferments, preferably obtained by autolysis of an albuminoid substance (*e.g.*, fish residue), or of brewers' or distillers' yeast. W. T. Lockett.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Isolation and properties of by-product in saccharin manufacture. W. Herzog (Oesterr. Chem.-Ztg., 1926, 29, 173—174).—In the saccharin mother-liquor of the alkaline oxidation of *o*-toluenesulphonamide a small amount of a very bitter-tasting substance

can be recognised (cf. "Chem. Tech. org. Verbindungen." Herzog, 1912, II, 507). Strong acidification of this mother-liquor yields a curdy precipitate containing, as well as some saccharin and *p*-sulphamidobenzoic acid, the bulk of this bitter substance. The filtrate from repeated boilings with water yields on slow cooling, white needles, m.p. (from water) 248°, $C_{14}H_{12}O_4N_2S_2$. A sodium salt of excessively bitter property is obtained by treatment with moderately dilute sodium hydroxide.

P. V. McKie.

Determination of arsenic in small quantities of organic compounds. O. Wintersteiner and H. Hannel (Mikrochem., 1926, 4, 155—167).—From 7 to 12 mg. of the compound are heated with 1 c.c. of 30 vol. % sulphuric acid and a few drops of concentrated nitric acid in a small Kjeldahl flask until copious fumes are evolved; 5—6 drops of perhydrol are added and heating is continued for a few minutes, after which the excess of peroxide is decomposed by two evaporations with 1 c.c. of water. If the substance contains iodine or bromine 0·3 c.c. of 4% potassium iodide solution is added and evaporation repeated to expel liberated iodine; re-oxidation with perhydrol and subsequent evaporation with water is then necessary. The acid is diluted with 1 c.c. of water and the solution transferred to a small flask, boiled to expel air, and cooled. The Kjeldahl flask is rinsed with previously boiled hydrochloric acid, and the rinsings are added to the main solution, which, after addition of 2 c.c. of 4% potassium iodide solution, is diluted to 20 c.c. The liberated iodine is titrated with 0·01*N*-thiosulphate delivered from a Pregl microburette, 5 drops of starch solution being added towards the end of the titration. A feeble rose tint is taken as the end-point. The results obtained are extremely good when the original substance does not contain bromine or iodine. The presence of these elements leads to slightly erratic results, even when the modified procedure mentioned above is adopted; without it, the results are much too high.

A. R. Powell.

De Myttenaere index for the chemical determination of the toxicity of arsenobenzenes. A. Contardi and U. Cazzani (Boll. Chim. farm., 1926, 65, 513—522).—In its latest modification, the method of determining the so-called D.M. index or the toxic arsenic in arsenobenzenes, is as follows: a solution of 1 g. of the product dissolved in 90 c.c. of boiling water is treated with 10 c.c. of dilute acetic acid and heated on a water bath, with occasional shaking, for 10 min. after precipitation commences. After being left for 10—12 hours in a cool place, the liquid is filtered, the filtrate heated to 60° with 5 c.c. of hydrochloric acid, and saturated with hydrogen sulphide, the temperature being kept at 60°. The liquid is filtered, the precipitate washed until neutral, and digested with 20 c.c. of ammonium carbonate solution. The filter is washed with water and the filtrate heated on a sand bath until the ammonia is eliminated, 10 c.c. of hydrogen peroxide and 10 c.c. of 50% sulphuric acid being carefully added and the liquid heated until white fumes are emitted. When cold the solution is again treated with 5 c.c. of hydrogen peroxide and evaporated. The cold liquid is treated cautiously with 10 c.c. of water and sufficient 1% permanganate solution to colour it permanently and then decolorised by 2% oxalic acid solution. The solution, mixed with 20 c.c. of 25% potassium iodide solution, is heated for 20 min. on a water bath, then cooled, and decolorised exactly with 0·1*N*-sodium thiosulphate solution. After addition of 25 c.c. of cold, saturated sodium carbonate solution and excess of sodium hydrogen carbonate, the liquid is titrated with 0·01*N*-iodine solution; the number of c.c. of this is the D.M. index, which should not exceed 12 (cf. B., 1924, 32). The authors' results show that the heating with acetic acid alters the novarsenobenzenes to an extent varying with their stability and with the nature of the salts present, and the precipitate formed by hydrogen sulphide may originate in this way. Since, also, *p*-hydroxy-*m*-aminophenylarsenious oxide does not respond to the above procedure, the authors consider that the D.M. index has no precise chemical signification (cf. Valeur and Launoy, B., 1925, 113). T. H. Pope.

Determination of total alkaloids, sugar, and oily substances in opium. J. N. Rakshit (Analyst, 1926, 51, 491—495).—*Total alkaloids.*—10 g. of opium are triturated with 50 c.c. of water for 30 min., the liquid is filtered, and the residue transferred to a mortar, with 50 c.c. of 4% hydrochloric acid, and ground for 15 min. After again filtering, the residue is extracted three times with 200 c.c. of ether and the united extracts are shaken with 50 c.c. of the dilute acid until the aqueous extract gives no precipitate with excess of sodium carbonate solution. The residue from the ethereal extract and all the filter papers is rubbed in a mortar with 25 c.c. of the dilute acid, the mixture filtered, and the process repeated until there is no precipitate with sodium carbonate solution after keeping for some time. The aqueous and acid extracts are treated with 25 g. of anhydrous sodium carbonate, added in very small quantities, and after keeping overnight the precipitate is filtered off, as in the B.P. process for morphine. After removal of the alkaloidal precipitate, the alkaline filtrate is evaporated to dryness, powdered, and repeatedly extracted with a boiling mixture of equal volumes of chloroform and absolute alcohol. Total alkaloids thus obtained varied from 35·0% in Hill opium from Kohat to 48·6% in a Chinese chandu sample. *Sugar* has a retarding effect on the precipitation of morphine, and is best determined by titration with Fehling's solution after removal of the bulk of the alkaloids. "*Added oil" or wax* is determined from a consideration of the figures for the proportions of natural wax, saponification value of the natural wax, and saponification value of the sample. Further, a natural raw opium containing varying quantities of added oily substances shows a fairly sharp gradation in the change of appearance when heated on a porcelain plate over steam. D. G. Hewer.

Examination of hydroaromatic alcohols and ketones. A. Noll (Z. Deut. Oel Fett Ind., 1926, 46, 689—690).—The determination of a ketonic group by means of hydroxylamine hydrochloride has been applied to the examination of commercial specimens of *cyclo*hexanol, the mixed methyl*cyclo*hexanols, and the corresponding hexanones. The boiling range and ketonic content of samples of these solvents of German manufacture are given.

E. Holmes.

Physiological assay of preparations of digitalis. E. Knaffl-Lenz (J. Pharm. Exp. Ther., 1926, 29, 407—425).—Various biological methods for the assay of digitalis preparations are described and discussed.

W. O. Kermack.

Manufacture of calcium citrate and citric acid from lemon juice. B. Melis (Giorn. Chim. Ind. Appl., 1926, 8, 467—469).—See B., 1926, 690.

Non-volatile constituents of the cotton plant. Power and Chesnut.—See XVI.

Patents.

Manufacture of acetic anhydride. H. Dreyfus (E.P. 257,968, 9.4.25).—Acetic anhydride may be prepared by passing acetic acid vapour over water-binding agents such as bisulphates or pyrosulphates (especially of the alkali and alkaline-earth metals), zinc chloride, etc., at 200—1000°, under reduced pressure if necessary. The acid vapour is led over or through the solid or molten agent, which may be spread on porous material. The agent may be continously regenerated by heating it in a chamber connected with the reaction chamber. The anhydride may be separated from the reaction gases by fractional condensation. B. Fullman.

Process for the manufacture of tartrates. Chem. Fabr. Dr. H. Stoltzenberg (E.P. 242,590, 6.6.25. Conv., 5.11.24).—Tartrates are obtained by halogenating neutralised fumaric acid or maleic acid, or a salt of the acids, and heating the resultant product in the presence of a carbonate or bicarbonate; or the product may be heated in an autoclave with carbon dioxide under pressure at 100°. A metallic catalyst such as bronze may be present in the treatment of the halogenated product. Two examples are given. B. Fullman.

Production of *iso*borneol from a mixture of camphene and associated oils. H. Gammay (E.P. 258,475, 4.2.26).—The mixture of pinene hydrochloride and accompanying hydrochlorides obtained by saturating pinene with hydrogen chloride is dechlorinated, and the resultant mixture of camphene and associated oils converted into a bornyl ester and hydrolysed. From the *iso*borneol the oil may be removed by centrifuging, the residual *iso*borneol being separated from the oil by distillation. B. Fullman.

Production of camphor from *iso*borneol. H. Gammay (E.P. 258,462, 7.1.26).—To *iso*borneol, subdivided in water (a small quantity of a non-oxidisable solvent being added if necessary) a mixture of sodium dichromate and dilute sulphuric acid is added. When the temperature rises to the m.p. of camphor, the reaction mixture is cooled and more chromic acid mixture again added, and so on. The sulphuric acid may be originally admixed with the *iso*borneol, sodium dichromate being added. B. Fullman.

Manufacture of esters of *iso*borneols and borneols. Chem. Fabr. auf Actien (vorm. E. Schering) (E.P. 250,555, 16.3.26. Conv., 11.4.25).—*iso*Borneol and borneol esters of greater purity than usual are obtained by treating camphene with excess of concentrated fatty acids in the presence of weak acids such as pyrophosphoric, boric, oxalic, and toluenesulphonic acids, or in the presence of strong mineral acids at temperatures substantially below 20°. For example, 10 parts of camphene, 20 parts of 95% formic acid, and 2 parts of pyrophosphoric acid are heated to 60°. The temperature rises to 70°, which must not be exceeded. In a few minutes the reaction is completed; the excess of formic acid is distilled off *in vacuo*, and the reaction product washed until neutral. The ester has a saponification value of 308, corresponding to 100% *iso*bornyl formate, with d_4^{20} 1·01. B. Fullman.

Production of symmetrical diarylguanidines. Silesia Verein Chem. Fabr. (E.P. 258,203, 28.5.26. Conv., 23.9.25).—*s*-Diarylguanidines are obtained almost quantitatively by treating the corresponding diarylthiocarbamides, suspended, together with metallic oxides, in organic solvents, with ammonia under pressure, at ordinary temperature. For example, s-*di-o-tolylguanidine*, m.p. 170°, is obtained in 92% yield by stirring 1 kg. of di-*o*-tolylthiocarbamide, 1·1 kg. of lead oxide, and 10 kg. of xylene, in the presence of ammonia maintained at a pressure of 3 atm., for 3 hrs., at about 20°.

B. Fullman.

Process of making aldols. C. J. Herrly, Assr. to Carbide and Carbon Chemicals Corp. (U.S.P. 1,598,522, 31.8.26. Appl., 22.5.24).—Acetaldehyde, or a higher homologue, contained in a closed vessel, is neutralised at 10° by adding dilute sodium hydroxide solution in a non-oxidising atmosphere, *e.g.*, nitrogen. The neutral aldehyde is then made slightly alkaline and maintained at 40° for nine hours by circulation of brine. After cooling to 20° dilute sulphuric acid equivalent to the total alkali used is added. The unconverted aldehyde is then distilled off *in vacuo* below 50°. An 83% yield is obtained, based on the aldehyde used. R. B. Clarke.

Nitrated ester. C. E. Burke and R. L. Kramer, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,598,474, 31.8.26. Appl., 18.9.24).—The patent relates to the preparation of esters of the formula $CHMe(ONO_2)\cdot CO_2R$, where R represents the radical of a monohydric alcohol containing more than three carbon atoms. These new compounds are almost insoluble in water and acids, soluble in organic solvents, good colloiding agents for nitrocellulose and of low volatility. *n*-Butyl nitrolactate is prepared as follows: *n*-Butyl lactate is first prepared by esterifying lactic acid with *n*-butyl alcohol in the presence of sulphuric acid. The ester is gradually added with vigorous agitation to a mixed acid of equal parts of sulphuric and nitric acids, the temperature being maintained at 15°. The mixture separates into two layers, the upper layer consisting of the nitrated ester, which is separated, washed, and dried. R. B. Clarke.

Process for producing 3 : 5-di(acetoxymercuri)-4-nitro-*o*-cresol and compounds thereof. Abbott Laboratories, Assee. of G. W. Raiziss (E.P. 236,538, 20.6.25. Conv., 2.7.24).—To minimise the formation of nitroindazole, 4-nitro-*o*-cresol is prepared by nitration of *o*-toluidine and diazotisation of the resultant 4-nitro-*o*-toluidine. The crude nitrocresol is dissolved in water

and sodium hydroxide and heated with mercuric acetate for 2 hrs. at 100°. The 3 : 5-*di(acetoxymercuri)-4-nitro-o-cresol* is separated as a brownish-yellow solid from the admixed mercury derivative of nitroindazole by extraction with alkali. It has germicidal properties. Its alkali metal salts are prepared by pouring its solution in the appropriate hydroxide into methyl alcohol and precipitating with ether. B. Fullman.

Manufacture of organic compounds of bismuth for therapeutic purposes. R. W. E. Stickings, and May & Baker, Ltd. (E.P. 258,641, 19.6.25).—Double salts of aliphatic hydroxy-polybasic acids (tartaric, citric, malic, etc.) with bismuth and organic bases (diethylamine, diethylaminoethanol, hexamethylenetetramine, piperazine, etc.) are prepared by adding an acid bismuth salt (such as bismuthyltartaric acid) to the base until solution is effected and a neutral product formed; or a neutral bismuth salt is treated with 1 mol. of base; or an acid salt of the base is treated with as much bismuth hydroxide as will dissolve, the resultant acid product being neutralised by further addition of base; or a neutral bismuth salt is treated with the corresponding salt of the base, the product when acid being neutralised with base, or the neutral product is obtained by carrying out the reaction in the presence of free base. The products are isolated by pouring the aqueous reaction solution into a non-solvent of the compound (alcohol etc.); or the solution may be stabilised for therapeutic use by addition of a reducing sugar such as glucose. These compounds have therapeutic advantages over the corresponding double salts of the acids with bismuth and the alkali metals. B. Fullman.

Manufacture of quinine salts of [hydr]oxyphenylarsinic acids. F. Hoffmann-La Roche & Co. A.-G. (E.P. 259,153, 9.6.26. Conv., 6.11.25).—Quinine salts of hydroxyphenylarsinic acids (*e.g.*, the p-*hydroxyphenylarsinate*, m.p. 177—178°) are prepared by reaction between quinine and the free acids, or between soluble quinine salts and salts of the acids. B. Fullman.

New complex alkali or alkaline-earth antimony compounds. W. Carpmael. From Farbenfabr. vorm. F. Bayer & Co. (E.P. 258,973, 17.7.25).—New complex alkali or alkaline-earth antimony compounds of trypanocidal action are obtained by treating the appropriate neutral salt of a mercaptocarboxylic acid with an oxygen compound of tervalent antimony; or by treating the free acids with the antimony compound and neutralising with an alkali or alkaline earth without isolation of the complex antimony acid produced (this method must be used in the preparation of derivatives of thioglycollic acid); or the complex acid may be isolated and afterwards neutralised. For example, *sodium antimony trithioglycollate*, $Sb(S\cdot CH_2\cdot COONa)_3$, is prepared by heating 20 parts of thioglycollic acid with 6 parts of freshly precipitated antimony trioxide and 50 parts of water for some time, filtering, neutralising with sodium carbonate, and evaporating to dryness. *Barium antimony trithioglycollate* and *sodium antimony thiosalicylate* are also described. B. Fullman.

Manufacture of new organic phosphorus compounds. A. J. Ransford. From L. Cassella & Co., G.m.b.H. (E.P. 258,744, 20.10.25).—9-Alkyl- (or benzoyl-) carbazolephosphinous acids (having antirachitic properties) are formed by the action of phosphorus trichloride on 9-alkyl-(or benzoyl-)carbazoles. Thus, melted 9-methylcarbazole is heated to 150° and phosphorus trichloride gradually added. The product, after cooling, is added to a mixture of sodium hydroxide solution (d 1·36), cooled with ice. After extraction with ether, the solution is concentrated and 9-*methylcarbazole*-3-*phosphinous acid* precipitated by addition of acid. 9-*Ethylcarbazole*-3-*phosphinous acid* is also described. The compounds are very resistant to acids. B. Fullman.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Contrast of photographic printing paper. L. A. Jones (J. Franklin Inst., 1926, **202**, 177—204, 469—516).—The psychophysical basis of contrast is discussed. Like subjective contrast, contrast as applied to photographic materials is a function of a rate or gradient factor and an extent factor, which are dependent respectively on the differential of density with respect to log E, and on the density scale available or used. In relative terms, contrast may be specified as the product of the two factors. The straight-line part of the characteristic curve of printing papers is generally so small that gamma is not a satisfactory specification of the gradient characteristic, and, in order to evaluate total contrast or contrast capacity, means for determining the mean effective value of $dD/d \log E$ are necessary. In practice the brightness scale of a subject rarely exceeds 250 and can in general be completely rendered on the straight-line part of a negative characteristic curve. This is not so with developing-out papers, and the entire density scale must generally be used. A statistical method of determining the portions of the positive material curve which can be utilised for reproducing a particular brightness scale is considered. As object a "typical landscape," in which the brightness contrast was 39 was used. Using a panchromatic plate which had just sufficient exposure latitude to render the object on the straight-line part of the characteristic curve, a group of nine negatives, equally exposed, was made, the development time being varied to give gamma values ranging from 0·38 to 1·32. The densitometric characteristics were determined on the negatives and on sensitometric strips developed with each negative. From the appropriate negatives groups of prints were made on 37 different developing papers, covering the entire range of contrast available in this type of material. From the prints on each paper judgment was made by 75 observers on the negative producing the optimal photographic quality. The data obtained are critically examined, and conclusions are drawn relating to the characteristics of the observers, the repeatability of judgments of print quality, and the average photographic quality considered as optimal by observers of different degrees of training in print judgment. The

gamma of the optimal negative for each paper is obtained, and from density measurements on the positives the density scale for optimal positives is obtained. These values form part of the data from which values of contrast for the positive materials will be computed.

W. Clark.

Comparison of metol-quinol and metoquinone developers. E. Muller (Bull. Soc. franç. Phot., 1926, 13, 226—229).—The experiments of Lumière and Seyewetz (B., 1925, 786) were repeated. Grinding together the metol and quinol of a metol–quinol developer before dissolving gives no increase in development rate. It is confirmed that the energy of a metoquinone sulphite developer is superior to that of a metol–quinol developer containing two mols. of metol and one mol. of quinol, with sodium carbonate equivalent to the sulphuric acid of the metol. If the carbonate content is doubled, a developer identical in properties with the metoquinone developer is produced. W. Clark.

Metoquinone developer. A. Hübl (Phot. Korr., 1926, 62, Mar. 31; Bull. Soc. franç. Phot., 1926, 13, 229—231, cf. preceding abstract).—The difference in development rate between metoquinone and metol–quinol found by Lumière and Seyewetz is confirmed. If the carbonate equivalent to the sulphuric acid of the metol in the metol–quinol formula is replaced by the equivalent of caustic soda, a developer identical in properties with the metoquinone formula is obtained. The carbon dioxide freed in the first developer probably decreases the activity of the developer. If the carbonate is doubled, bicarbonate is formed, and the developer is as active as the metoquinone formula. Addition of 0·5% of bicarbonate to the metoquinone developer does not affect its actitivy. Quinol can be eliminated from the metol–quinol formula containing carbonate equivalent to the sulphuric acid of the metol without affecting its developing power. A metoquinone developer can be regarded merely as a metol developer. Only if alkali is added does the quinol become active. Metoquinone can in all cases be replaced by metol–quinol. Metoquinone without added alkali corresponds to neutralised metol, and alkaline metoquinone to a normal metol–quinol developer. W. Clark.

Patents.

Photographic copying. A.-G. f. Anilin-Fabr. (E.P. 243,023, 16.11.25. Conv., 14.11.24).—Printing surfaces and preliminary copies are prepared by the reflection copying method in such a way that the picture is formed on the layer and not within it. The sensitised colloid layer may be converted into a flat printing surface by development and treatment as in preparing a collotype printing surface or the printing surface may be inked with a fatty ink and then sprayed, after drying, with bronze powder etc. An exposed sensitised colloid layer which is soluble in water or which swells in presence of water, is inked with a fatty ink and then developed with water containing, if necessary, lactic acid etc. to assist in development. After exposure and moistening, a sensitised hygroscopic colloid layer is sprayed with fine powder. A sensitive silver halide emulsion may be used and developed either physically or chemically with intensification by physical means.

W. Clark.

Pictures by the dye-transfer process. R. von Arx (E.P. 245,163, 24.12.25. Conv., 24.12.24).—Pictures are produced by converting a positive silver image into a mordant, *e.g.*, of copper silver ferrocyanide, dyeing with a dye having an affinity for this mordant, and printing from this on to a simple layer, such as of collodion, or a layer carrying a mordant, *e.g.*, gelatin cuprous thiocyanate, the affinity of which for the dye exceeds that of the positive mordant. Polychrome images are produced by successive printing from a set of mordant colour selection positives, or from positives prepared by the selective colour method from negatives made with multicolour screens and fixed or treated in dilute acid. The mordant may be removed after printing by an agent not affecting the colours. W. Clark.

Photographic reversal processes. Kodak, Ltd. From Eastman Kodak Co. (E.P. 258,814, 11.5.26).—Re-reversal in the second development in the production of direct positives by reversal (cf. E.P. 176,357 and 218,632) is prevented by the addition of soluble silver salts to the bleaching solution in order to eliminate by precipitation the effects of harmful impurities, such as halogen salts, which may be present. A suitable bleaching solution consists of dilute solutions of potassium permanganate, sulphuric acid, and silver nitrate.

W. Clark.

Kinematograph pictures in natural colours. F. Lierg and K. Pokorny (E.P. 245,470, 2.1.26. Conv., 2.1.25).—A three-colour image is formed by using a positive film projected through two of the colours in the form of screens, and itself toned or dyed the third colour, either on one or both sets of pictures. The colour screens may be modified from being complementary to the taking screens, and the colour of the toned silver image may be modified, in order to obtain a truer rendering on projection. The filter colours may be formed on the film itself by providing one set of component film pictures with a waterproof protective coating bearing the respective filter colour, the other set being dyed by immersing the whole film in the complementary colour. By hardening the gelatin portions containing the coloured silver image by a suitable composition of the toning or developing bath, on subsequent dyeing the colouring is stronger in the clear parts of the gelatin film than in the portions containing the silver image. W. Clark.

Photographic film for colour photography. R. Berthon, Assr. to Société du Film K.D.B. (U.S.P. 1,600,923, 21.9.26. Conv., 4.12.22).—The emulsion is coated on a support having its outer face formed with a net of microscopic lenticular elements, in number more than 500 /sq.mm., so as to produce diffraction in addition to refraction. W. Clark.

XXIII.—SANITATION; WATER PURIFICATION.

Determination of toxic substances in insecticides etc. I. Volumetric determination of thallium in "Zelio" preparations. J. Bodnár and A. Terényi

(Z. anal. Chem., 1926, **69**, 29—36).—Thallium may be determined volumetrically in "Zelio" preparations (rat poison) by dissolving the thallous chromate obtained by the method of Mach and Lepper (B., 1926, 320) in hydrochloric acid and potassium iodide and titrating the liberated iodine but, owing to the time consumed in the preliminary operations, the method is slow. A much more rapid, although not quite so accurate procedure consists in carbonising the paste at as low a temperature as possible, extracting the thallium from the powdered residue by boiling with 50 c.c. of 10% sulphuric acid, adding 2—3 c.c. of 10% hydrochloric acid, and titrating the thallium with permanganate at 70—90°. The solution is standardised against pure thallous sulphate under the same conditions. The results obtained for thallium should be increased by 0·04%.

A. R. Powell.

Chemical characteristics of some trade wastes. A. M. Buswell, R. E. Greenfield, and R. A. Shive (Ind. Eng. Chem., 1926, **18**, 1082—1084).—Wastes produced in a pea cannery, in strawboard, paper, and roofing factories, in two distilleries, and in a corn cannery have been examined and shown to consume from 10 to more than 100 times as much oxygen per unit as is consumed by ordinary sewage. D. G. Hewer.

Treatment of packing house, tannery, and corn products wastes. F. W. Mohlman (Ind. Eng. Chem., 1926, **18**, 1076—1081).—The methods used in Chicago for combating the effect of the major industrial wastes are evaluated, together with results of experimental treatment of the water for the last 15 years. The activated sludge process is recommended for packing-house wastes, preliminary screening and settling in a Dorr clarifier and subsequent mixing with large volumes of domestic sludge for tannery wastes, and trickling filters for corn products wastes. D. G. Hewer.

Disposal of some organic trade wastes. E. Bartow (Ind. Eng. Chem., 1926, **18**, 1085—1086).—For packing-house wastes the activated sludge process is regarded as best, unless there is a chance of high dilutions; for beet sugar factories the greatest problem is the disposal of the Steffens waste, from which potassium chloride, potassium sulphate, potassium nitrate, betaine hydrochloride, and glutamic acid have been obtained. For starch wastes, concentration of the wastes should be reduced within the plant and subsequent treatment in a sprinkling filter type of plant is recommended.

D. G. Hewer.

Apparatus for measuring turbidity and colour of water. W. Olszewski (Chem.-Ztg., 1926, **50**, 694).—The water under examination in a tube 20 cm. long is compared with distilled water in a similar adjacent tube by means of a half-shade photometer. The colours of the two are matched by the insertion of suitable coloured glasses, while the relative turbidities, against Ostwald's normal white, are determined by varying the amount of light passing through the distilled water by means of a graduated slit. F. R. Ennos.

Effect of *X*-rays of long wave-length on *B. prodigiosus*. J. J. Trillat (Compt. rend., 1926, **183**, 614—616).—*X*-Rays of long wave-length ($\lambda = 2·23$ A.) render cultures of *B. prodigiosus* sterile in 1—2 hrs. with direct irradiation; with oblique irradiation, colonies on the edge of the beam of rays showed increased growth.

L. F. Hewitt.

Rôle of the chemist in the investigation of problems of industrial hygiene. C. A. Klein (J. Oil Col. Chem. Assoc., 1926, **9**, 270—290).—Presidential address, delivered on October 22.

Patents.

Treatment of sewage. K. Imhoff (E.P. 255,833, 17.6.26. Conv., 22.7.25).—Sewage is treated in settling basins containing colloid collectors (consisting of light matters, *e.g.*, coarse wood wool, held together by a wire or wickerwork net) by imparting to the sewage, by means of air forced from below the colloid collectors, a circulating current upwards through the collectors and downwards in the free spaces of the settling basin laterally thereof. Accumulated sludge may be removed from the colloid collectors from time to time by increasing for a short period the supply of air, and with a large breadth of colloid collector the air supply pipe may be moved to and fro over the bottom. W. T. Lockett.

Antiseptic. G. E. E. Crosnier (E.P. 258,058, 12.8.25).—A chemically neutral, odourless, non-toxic and readily soluble antiseptic and deodorant is prepared by mixing together zinc phenolsulphonate, preferably the *p*-compound, a second zinc salt, preferably zinc sulphate, and an acetate, *e.g.*, sodium acetate. W. T. Lockett.

Disinfectants. A. Brick (E.P. 258,110, 29.10.25).—A disinfectant in solid form is prepared as follows:—Rice starch is soaked in a solution of potassium permanganate and subsequently dried and powdered. The powder is mixed with a deodorising composition consisting of, for instance, oil of turpentine, pine-needle oil, musk, terpineol, lemon-grass oil, geranium oil, camphor oil, and juniper berry oil and to the pulpy mass obtained are added kieselguhr, rice flour, soap flakes, and soap powder and finally, paraformaldehyde. The mass is then dried and pressed. W. T. Lockett.

Disinfectant composition. G. G. Gordon (E.P. 258,678, 29.6.25).—One or more oils or extracts of the eucalyptus species are mixed with an oleaginous substance (*e.g.*, oleic acid) or resin until the latter is dissolved. An aqueous alkaline solution containing 2½% of sodium hydroxide is then added until the eucalyptus oil content approximates 37% of the whole. W. T. Lockett.

Fumigant. G. H. Buchanan (E.P. 258,324, 13.6.25).—The fumigant consists of cyanogen chloride or a mixture of cyanogen chloride and hydrocyanic acid in liquid form, prepared so as to give a slightly acid indication with methyl orange to prevent decomposition. A small amount of cyanogen chloride or a mixture rich in cyanogen chloride is first introduced into the space to be fumigated, to act as a warning to persons who may be present, and then the proper amount of the fumigant of the desired composition. W. T. Lockett.

Vermin-killer. E. Derrégibus (E.P. 258,953, 2.7.25).—A mixture of coal tar, methyl salicylate, and naphthalene.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DECEMBER 24, 1926.

I.—GENERAL; PLANT; MACHINERY.

Principles of modern specifications for boiler feed-water. A. Splittgerber (Z. angew. Chem., 1926, **39**, 1340—1345).—The necessity of careful analytical control of boiler feed-water, before and after softening, for efficient and economical running, is emphasised, and the methods of softening are briefly described. The alkalinity of the water in the boiler should never be below 0·4 g. of sodium hydroxide or 1·85 g. of sodium carbonate per litre to prevent the corrosive action of any nitrates present, neither should it exceed about 3 g. of sodium carbonate or 2 g. of sodium hydroxide per litre or there is danger of the development of fissures around the rivet holes. The presence of sodium sulphate equivalent to 1—3 times the weight of carbonate present retards the action of the alkalis on the steel, but in no case should the total salt content of the boiler water exceed 20 g. per litre. The residual hardness of the feed-water should not be greater than 2°. The simultaneous presence of more than 5 mg. of oxygen and 35 mg. of carbon dioxide per litre of feed-water leads to severe corrosion; such water should therefore be de-gassed before use. This applies particularly to water softened by the permutit process when the water had originally a high carbonate hardness. A. R. Powell.

Caustic embrittlement of steel. Parr and Straub.—See X.

Patents.

Furnaces. Trocknungs-, Verschwelungs-, und Vergasungs-Ges. m.b.H., L. Honigmann, and F. Bartling (E.P. 259,389, 9.10.25).—A furnace for the heat treatment (distillation) of pulverulent or pulpy material is constructed of a rotary annular hearth —preferably supported by floating in water, the water space being sealed off from the heating space above by sand seals. In operation, one sector of the hearth is preheated, and, after charging the material, the heating may be continued on the other sector. At the end of the treatment the material is preferably removed from the hearth by a blast of gas, and may pass out of the furnace with the gases of combustion, being separated elsewhere and part of the gases returned to the furnace. The upper surface of the hearth may be ribbed and means provided for spreading the material in a thin layer. B. M. Venables.

Pulverisers. Syracuse Pulverizer Corp., Assees. of A. J. Briggs (E.P. 256,562, 6.10.25. Conv., 8.8.25).—A disintegrator of the hammer type is surrounded by another of the cage type, the first, third, etc. cages being stationary, and the second, fourth, etc. rotating. The cage bars are closely spaced, and are shaped like a keystone in section. The material (*e.g.*, coal) is drawn through the apparatus by a fan, and a regulator for the air stream is interconnected with a regulator for the coal feed so that the proportion may always be correct for combustion. B. M. Venables.

Pulverising mill. A. M. Read (U.S.P. 1,598,933, 7.9.26. Appl., 9.10.20).—A rotary barrel is provided with transverse screens dividing it into separate grinding chambers. The screens are curved spherically about centres in the approximately horizontal axis of rotation. H. Holmes.

Centrifugal separators. T. C. Thomsen (E.P. 259,514, 21.6.26).—A centrifugal bowl for removing suspended solids from liquids is provided with an inserted device which has a scraping ring on the bottom to remove the solid matter when the insertion is raised out of the bowl. The insertion also has radial walls dividing the bowl into larger segmental compartments for separation, and smaller segments which permit easy overflow of the clear liquid; ports communicating between the larger and smaller compartments are formed in the lower part of the radial walls, so that although the clear liquor flies off the upper rim of the bowl as usual, it leaves the separated solids in the lower part of the bowl. B. M. Venables.

Centrifugal separating machine. H. Feldmeier, Assr. to D. H. Burrell & Co., Inc. (U.S.P. 1,600,328, 21.9.26. Appl., 4.12.22).—The bottom of the rotary separating bowl is provided in its under side with a central discharge throat cavity, opening downwards and surrounding the vertical driving spindle. The lower end of an open-bottomed shell is seated removably on the bottom of the bowl. Liquid to be separated is fed into the upper portion of the bowl through an opening outside of a hollow hub portion closed at its upper end, and seated removably on the upper end of the spindle. Liquid discharge passages lead through the bottom of the bowl into the discharge throat cavity, and at least one such passage extends inwards from its point of communication with the interior of the bowl. A receiving pan is provided with a central bottom opening surrounding the cavity above its lower end, and a discharge passage leads from the cavity above the bottom of the pan. H. Holmes.

Evaporating organic solvents. H. Bollmann (E.P. 259,497, 13.5.26. Conv., 5.1.26).—Organic solvents are evaporated from liquids containing them by atomising them by steam under pressure and injecting the mist tangentially into a cylindrical vessel, the solvent being removed as vapour, then condensed, and separated from the water by centrifugal means. The object of the steam atomisation is to avoid bumping, raised b.p., etc. at the end of the evaporation, so that the preliminary stages

of the evaporation may be effected by ordinary means, and the spraying vessel may be heated and operated under reduced pressure. The distilling operation may be repeated as often as desired. B. M. VENABLES.

Apparatus for treating liquids with gases. W. E. GREENAWALT (U.S.P. 1,598,858, 7.9.26. Appl., 2.4.23). —The liquid is contained in a tank provided, in its lower portion, with oppositely-positioned injectors, delivering into the body of the liquid a mixture of gas and liquid in horizontal streams. The liquid inlets and outlets of the injectors communicate with the tank, and means are provided for supplying gas to the injectors under pressure. H. HOLMES.

Processes for the separation of the constituents of ternary gaseous mixtures. AIR REDUCTION CO., INC., Assees. of C. C. VAN NUYS (E.P. 255,104, 12.7.26. Conv., 11.7.25).—A ternary gas mixture, particularly air, is separated into its three constituents, oxygen, nitrogen, and argon, by liquefaction and rectification in a main column, which delivers oxygen and nitrogen. From the main rectifier a liquid rich in argon is drawn off and subjected to further rectification in an auxiliary column, of which the upper part is cooled by the nitrogen from the main rectifier and the lower part heated by the exhaust from the expansion engine used in the liquefaction of the original air and/or the mixture of gas and liquid from the liquefier. The liquid from the auxiliary rectifier (mainly oxygen) returns to the main rectifier and cools the original incoming air. B. M. VENABLES.

Apparatus for treating corrosive gaseous fumes. M. M. MEDIGOVICH (U.S.P. 1,599,027, 7.9.26. Appl., 26.5.24).—The upper portion of a furnace stack is connected by a downwardly-inclined flue to an inlet in the lower end of a tank containing water, and provided with a top outlet. A pocket is provided in the lower wall of the flue for receiving solids gravitating within the flue. The pocket communicates by a valve, normally closed, with a pipe leading to a separating trough. Means are provided within the flue for producing a forced downward circulation of fumes into the water in the tank, for spraying water into the stack and flue, and for supplying water to the pocket to maintain a predetermined level therein. H. HOLMES.

Packing powdered material. C. PONTOPPIDAN (E.P. 259,299, 7.7.25).—By mixing powders with air at any desired temperature and pressure, the material, as regards movability, will exhibit the fluidity of liquids, and thus may be poured into containers. In the case of cement, the addition of air sufficient to increase the total volume by 15—20% enables it to be run into receptacles by gravity. F. G. CROSSE.

Heating liquids by introduction into molten metal. D. RIDER and J. S. WATTS, Assrs. to THERMAL INDUSTRIAL AND CHEMICAL (T.I.C.) RESEARCH CO., LTD. (U.S.P. 1,600,139, 14.9.26. Appl., 28.2.24. Conv., 7.4.23).—See E.P. 218,723; B., 1924, 735.

Evaporator. H. FOTHERGILL (U.S.P. 1,600,106, 14.9.26. Appl., 2.8.24. Conv., 24.8.23).—See E.P. 225,271; B., 1925, 86.

Centrifugal separator. H. THOMASSEN (U.S.P. 1,599,502, 14.9.26. Appl., 11.11.24. Conv., 27.5.24).—See E.P. 233,608; B., 1925, 540.

Deflocculating solids. G. W. ACHESON (E.P. 259,889, 23.7.26. Conv., 8.3.26).—See U.S.P. 1,595,052; B., 1926, 857.

Thermostats. P. M. SALERNI (E.P. 259,680, 16.7.25).

Hydrometers. P. STEVENSON and P. STEVENSON, JUN. (E.P. 258,125, 20.11.25).

Furnaces. A. RYNER (E.P. 258,486, 4.3.26).

Reversal and control of regenerative furnaces. A. E. WHITE. From MORGAN CONSTRUCTION CO. (E.P. 259,801, 23.12.25).

Exchangeable furnace arch. E. JÜRGES (E.P. 259,725, 29.8.25).

Liquid and air or other gas separator. H. D. BINKS (E.P. 242,603, 15.9.25. Conv., 10.11.24).

Absorption refrigerating apparatus. L. REEHSING (E.P. 260,161, 26.3.26).

Determining the volume and weight of the solid or liquid contents of receptacles. BRITISH LAUNDERERS' RESEARCH ASSOC., R. G. PARKER, D. N. JACKMAN, and J. N. VOWLER (E.P. 259,662, 13.7.25).

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Spontaneous combustion of coal: the most readily oxidisable constituents of coal. W. FRANCIS and R. V. WHEELER (Safety in Mines Res. Bd., Paper No. 28, pp. 51; cf. A., 1925, i, 373).—A further study has been made of the products obtained when coal is oxidised in a current of air at 100° and 150°. The coals used were the vitrain, durain, and fusain portions respectively of the Nottinghamshire Top Hard seam and the clarain portion of the South Yorkshire Parkgate seam, which contains pyritic inclusions. A slow current of moist air was passed over the coal, ground to 60—90 mesh, for five months at 100°, and for a further six months at 150°. Small quantities of formaldehyde, methyl alcohol, and acids were evolved during the first three months, but no acetone, acetaldehyde, or ethyl alcohol could be detected. Proximate analyses, ultimate analyses, determinations of loss in weight, of calorific value, of the compounds soluble in pyridine, and in pyridine and chloroform, and of the ulmins soluble in alcoholic potash, were made at intervals. Oxidation at 150° transformed nearly the whole of the bright coals (95·5% of the Top Hard vitrain and 90·5% of the Parkgate clarain), but only part of the dull coals (46·0% of the durain and 24·0% of the fusain) into ulmins soluble in alkali. Further oxidation, even at a higher temperature, had little effect on the residue. The constituents of a coal are divided into "reactive" compounds, which are those convertible into alkali-soluble ulmins by mild oxidation, and "inert" compounds, comprising the plant entities which remain insoluble after complete conversion of the ulmins into the soluble form. The "reactive" compounds are responsible for the spontaneous combustion of coal. The ulmin portion of several bituminous coals has been examined chemically. Analysis of the insoluble copper salt of the regenerated ulmins gives a value for the equivalent of approximately 170. The hydroxyl content is 2·5%, and the nitrogen content 2·2—2·25%, the

latter occurring principally in a closed ring. Oxidation with dilute nitric acid gave aliphatic acids (acetic, oxalic, etc.), aromatic acids (benzenetetracarboxylic etc.), and nitrophenols (picric acid etc.). It is concluded that the nuclei of the ulmin molecules consist of compact systems of six-membered annular groupings connected together by heterocyclic structures such as pyrrole and furan. The general characters of the various plant entities that can be isolated from bituminous coal are briefly described. A. B. Manning.

Pyritic oxidation in relation to the spontaneous combustion of coal. H. Macpherson, N. Simpkin, and S. V. Wild (Safety in Mines Res. Bd., 1926, Paper No. 26).—The different forms in which pyrites occurs in coal are classified, and the location of their occurrence in the Lancashire coalfield (Ravine Seam) is described. The oxidation of pyrites in coal causes an increase in the quantity of iron and sulphur soluble in hydrochloric acid. This fact has been made use of to determine the rate of oxidation of such pyritic matter. In certain portions of the seam the oxidation proceeded rapidly, whereas in others exposure to the atmosphere for one year made little alteration to the state of oxidation. Massive pyrites, occurring in bands covering extensive areas, was stable in some cases, but in others oxidised and disintegrated rapidly. Nodular pyrites occurring in coal in round or oval masses oxidised rapidly. Granular pyrites, present either as isolated grains or as segregated masses, resisted oxidation. Comparison of laboratory results with those from observed pyritic oxidation occurring in the pit showed close agreement. S. Pexton.

Relation between the physical condition of coal samples and their adsorptive power. D. J. W. Kreulen (Brennstoff-Chem., 1926, **7**, 331—332).—The relative adsorption of ferric hydroxide from a solution of ferric chloride by different coals varies with the degree of subdivision of the samples. The suggested method of classifying coals based on the amount of this adsorption (Stadnikov and Proskurnina, B., 1926, 729) requires, therefore, accurate standardisation of the size of the particles used. The differences observed by Stadnikov and Proskurnina may have been due in part to variations in the distribution of the differently-sized particles in the different types of coal. A. B. Manning.

Relation between coal analysis and carbonisation products. S. Qvarfort (Gas J., 1926, **176**, 33—36). —Various classes of coal, ranging in volatile content from 4·7 to 33·6% on the organic basis, were carbonised in an experimental retort setting comprising two horizontal retorts each carrying a charge of approximately 2·5 cwt. The combustion chamber temperature was 1200°. The quality of coke obtained was not comparable with that produced from the same coal carbonised in larger installations. The yield of coke does not appear to bear any simple relation to the volatile content of the coal from which it is produced: *e.g.*, on an organic basis, anthracite breeze containing 4·7% of volatiles yielded 88·4% of coke; American coking coal containing 17% of volatiles yielded 84·6% of coke; Durham gas coal containing 29·5% of volatiles yielded 75·3% of coke; and Yorkshire gas coal containing 33·6% of volatiles yielded 71·5% of coke. Empirical relations between the volatile content of the coal, the gas yield, and the calorific value of the gas are represented graphically. S. Pexton.

Combustibility and structure of cokes [from brown coals]. P. Niemann (Brennstoff-Chem., 1926, **7**, 325—331).—120 g. of dry coke (2—3 mm. size) were slowly heated in a current of air; on ignition, the source of heat was removed and the subsequent variation of the temperature of the coke was observed. The temperature–time curves permit an estimate being made of the "combustibility" of the coke. Cokes exhibiting a rough dull-black surface and giving obtuse-angled particles burn more readily than those with a silver-grey graphitic surface and sharp-angled fracture. The combustibility is greatly influenced by the crystallisation of salts on the surface and in the pores of the coke. Removal of these salts by washing is not possible on the large scale; quenching with water or incomplete washing of the coke may result in an increased surface crystallisation and, therefore, decreased combustibility. The ignition temperature falls with the fineness of subdivision of the coke; the combustibility, however, under the conditions of experiment is a maximum for particles in the neighbourhood of 1—2 mm. size. When the porosity falls below 35% the coke burns with difficulty. The combustibility is a better criterion of the practical heating value of a coke than is the calorific value. A. B. Manning.

Studies in carbonisation. I. Influence of size of coal. 16th Report of Gas Investigation Comee. Inst. Gas Eng. (Gas J., 1926, **175**, 802—826).—The investigation of a Nottinghamshire gas coal in an experimental carbonisation apparatus consisting of a horizontal circular retort of nickel–chromium–iron alloy carbonising 30 lb. of coal is described. The gases are drawn off at the bottom of the retort mouthpiece and, after passing through air and water coolers, are purified from ammonia by dilute sulphuric acid and from hydrogen sulphide by caustic soda solution. By suitably gearing two meters a proportionate sample of the gas made is collected in a sample gas holder and an automatic recorder indicates the rate of gas production. Coal in 5 grades, viz., 1¼″—¾″, ¾″—½″, ½″—¼″, ¼″—10 mesh, 10—30 mesh were carbonised at 978° for 2¾ hrs. The thermal value of the gas in therms (gross)/ton of coal increased from 72·3 in the largest size coal to 75·8 in the smallest. This was accompanied by a reduction in the tar yield from 8·3 to 6·2 gal./ton. The rate of gas production was greater for the larger sizes of coal in the early stages of carbonisation, but the total volume of gas evolved at the end of 2¾ hrs. was greater for the smaller grade coal. An increase in the temperature of carbonisation produced an increase in the rate of gas evolution in the early stages; a diminution in the time of carbonisation to 2 hrs. diminished the gaseous products of carbonisation. The structure of the coke produced is shown by a number of photographs of macro- and micro-sections, set in plaster and polished. The coke from the 10—30 mesh coal is more uniform in structure than that from the larger grades; the

walls of the larger cells contained a greater number of small pores. A. C. MONKHOUSE.

Influence of ash constituents in carbonisation and gasification of coal. II. C. B. MARSON and J. W. COBB (Gas. J., 1926, **175**, 882—891).—Special cokes were made by adding 5% of silica, alumina, lime, and ferric oxide and equivalent quantities of calcium carbonate and sodium carbonate to separate portions of a coal containing less than 1% of ash (cf. B., 1925, 620). The behaviour of these cokes at 1000° on gasification in steam at rates of 1—21 litres/hour was compared with that of the "pure" coke as previously described (B., 1924, 659). The additions of silica, alumina, and fireclay gave similar results to the "pure" coke but the calcium oxide, ferric oxide, and sodium carbonate showed a marked increase in reactivity. At 10 litres/hour the steam decomposed was 61% for the "pure" coke, 82%, 91%, 98% for the calcium oxide, ferric oxide, and sodium carbonate cokes, respectively, whilst the corresponding percentages of carbon dioxide in the water-gas made were 9·2, 5·4, 2·6 and 0·4. Additions of 1—5% of iron oxide and 0·5% of sodium oxide (as sodium carbonate) also gave increased gasification. It was deduced from the increased reactivity obtained by impregnating a portion of the "pure" coke with sodium carbonate solution that the reactivity is chiefly due to a specific catalytic effect of the added compound and not to the alteration in physical structure on carbonisation resulting from the addition. Similar increases in reactivity were observed at 850° using carbon dioxide; the percentages of carbon monoxide for the same rate of carbon dioxide were 6·6, 29·9, 45·6, and 89·0 for the "pure," iron oxide, calcium oxide, and sodium carbonate cokes, respectively. A. C. MONKHOUSE.

Examination of products of combustion from typical gas appliances. I. 15TH REPORT OF GAS INVESTIGATION COMEE. INST. GAS. ENG. (Gas J., 1926, **175**, 787—801).—A detailed examination of the iodine pentoxide method for the determination of small amounts of carbon monoxide is described (cf. J., 1926, 164 T). The gas to be examined is drawn by aspirators at the rate of 1 cub. ft./hour, through a train of apparatus consisting of calcium chloride tubes, potash bulbs, phosphorus pentoxide tube, a preheater, and an iodine pentoxide tube heated at 120° in an electric furnace. 80 g. of iodine pentoxide are used and the iodine evolved is absorbed in 10% potassium iodide solution. The apparatus is first swept through with air already passed through a similar train of apparatus. The iodine absorbed is titrated with N/500-sodium thiosulphate solution using starch as an indicator. The importance of standardising the conditions of titration is emphasised. The influence of town gas, hydrogen, methane, unsaturated hydrocarbons, benzene, oxides of sulphur and nitrogen on the determination is examined. Carbon monoxide in gases containing less than 50 parts per million can be measured with an accuracy of 1—2 parts. The air of Leeds in summer had a carbon monoxide content of 1—2 parts per million and in winter about 5 parts. Experiments on the aeration of a 1000 c.-p. Keith high-pressure lamp showed the maximum air : gas ratio at 68 in. pressure burning 18 cub. ft./hr. of gas of calorific value 472 B.Th.U./cub. ft. was 4·58 with the air regulator fully open. The maximum illuminating power was attained by cutting down the primary aeration to 3·48, the theoretical being 3·91 cub. ft. of air per c. ft. of gas. A. C. MONKHOUSE.

Recording gas calorimeter of the flow type. S. W. PARR (Ind. Eng. Chem., 1926, **18**, 1194).—The apparatus consists primarily of two similar vessels. Water flows into one and gas into the other. By the operation of suitably arranged valves, gas then displaces the water in the one and water the gas in the other. The gas thus displaced passes to a burner and communicates its heat of combustion to the water displaced. The temperature change in the water is thus brought about by an equal volume of gas, and a factor thus obtains which varies directly with the calorific value of the gas. The temperature difference is utilised to actuate a suitable recording mechanism. The rise and fall of the water level in the vessels actuates the contacts for operating electrically the change-over valves. R. A. A. TAYLOR.

Distillation of petroleum with gases. A. DOBRJANSKI and K. MATUSSOVSKI (Neftjanoe Chozjajstvo, **10**, 236—242; Chem. Zentr., 1926, II, 303).—A gas is passed through 400 c.c. of topped petroleum at a temperature of 250°, at a velocity of 20 c.c./sec. till 80 c.c. are distilled off, and then at 50 c.c./sec. till a further 21·5 c.c. are collected. In distillation in a stream of coal gas (d 0·47) for 33—34 min. the distillate had d 0·83125, flash point 38·5°, iodine value 2·6. In distilling with air for 20 min. the distillate had d 0·8327, flash point 42°, iodine value 2·7. In distilling with carbon dioxide for 18 min. the distillate had d 0·8318, flash point 39·5°, iodine value 2·25, and in distilling with hydrogen the distillate had d 0·8312, flash point 39·5°, and iodine value 2·39. The time of distillation is proportional to the square root of the density of the gas used. The rate of distillation is not constantly proportional to the speed at which the gas is passed. H. MOORE.

Corrosion in petroleum distillation. H. SCHMIDT (Petroleum, 1926, **30**, 1139—1140).—Corrosion in condensers is due to the presence of hydrogen sulphide from the oil and hydrochloric acid from magnesium chloride in the water which accompanies the oil. No satisfactory resistant alloy has been found. Nickel-chrome or rustless steel and block tin gave the best results, and wrought iron and steel the worst. The risk of corrosion by hydrogen sulphide in cracking plants at high temperatures is mentioned. W. N. HOYTE.

Refining transformer and turbine oils. N. BUTKOV (Neftjanoe Chozjajstvo, **10**, 388—392; Chem. Zentr., 1926, II, 305).—To identify the antioxidising agent of the petroleum resins, oxygen was passed at 120° through a transformer oil to which naphthalene, phenol, aniline, α-naphthol, anthracene, and α-naphthylamine had been added. The first three substances had little effect, but the last three had a strong antioxidising action. The presence of 1·5% of α-naphthylamine reduced the acidity after passing oxygen for 60 hrs. from 21·05 to 0·18 mg. of potassium hydroxide. If the acid constituents be removed from acid-refined oil by

silica gel instead of alkali, the stability of the oil is improved owing to the absence of sodium salts of the organic acids. The tendency of the paraffin wax to oxidise is increased by treatment with strong alkaline solutions after oleum refining. The treatment of the oil with silica gel occupies 1·5 hrs. The gel absorbs 1% of sulphonic acids, which may be recovered, and the gel may be regenerated by heating. It is proposed to substitute sulphuric acid in refining by the oxygen of the air. On passing air at 100° for 12 hrs. and treatment with 20% of silica gel a light oil of *d* 0·885 and acidity 0·02 was obtained. On passing oxygen at 120° for 36 hrs. the acidity was 0·03. Lead vessels cannot be employed. An increase of pressure of the air to 2 atmospheres shortens the process by 3 hrs. Turbine oil treated by this process is less liable to emulsify.

H. Moore.

Formolite values of Grozny petroleum. B. Tarassov and V. Rudenko (Neftjanoe Chozjajstvo, 1926, **10**, 245—247; Chem. Zentr., 1926, II., 308).—Little concordance is shown by the formolite values determined by various experimenters, owing to the varying rapidity of the addition of formaldehyde and local overheating. If the first is normalised and the second avoided by dilution with low-boiling aliphatic benzine concordant figures are obtained. For the fractions up to 100° (20 c.c.), 100—150° (10 c.c.), and 150—200° (5 c.c.), 20 c.c. of sulphuric acid and 15 c.c. of formalin are taken, with the addition of benzine in the last case. The 200—250° fraction (5 c.c.) is treated as the 150—200° fraction, and the 250—300° fraction (5 c.c.) is treated with 25—30 c.c. of acid, 20—25 c.c. of formalin, and 15 c.c. of benzine. For these fractions of a wax-free oil the values 5·7, 12·0, 20·7, 24·4, and 31·0 were found respectively, and for wax-containing oil 5·0, 11·4, 18·0, 20·2, and 21·4.

H. Moore.

Determination of hard asphaltum in dark mineral oils. D. Holde (Petroleum, 1926, **22**, 799—800).—The asphalt content of two cylinder oils was determined by (A) precipitation with light petroleum, dissolving the precipitate in benzene, evaporating, and repeated boiling with absolute alcohol to remove paraffin wax; (B) the same procedure and, after each boiling with alcohol, redissolving the precipitate in benzene, evaporating, and extracting with alcohol till the redissolved precipitate shows no traces of paraffin wax. A cylinder oil giving the value 0·19—0·24% by method (A) gave the value 0·1—0·11% by method (B), and the results were more concordant. H. Moore.

Chemistry of gasolines, particularly with respect to gum formation and discoloration. B. T. Brooks (Ind. Eng. Chem., 1926, **18**, 1198—1203).—Further research is described on the colour and the resinifying and gum-forming propensities of petrols, cracked light oils, and paraffins. The olefines on atmospheric oxidation form peroxides which break down to give aldehydes (including formaldehyde), ketones, water, carbon dioxide, and simple organic acids. The peroxides and liquid aldehydes and ketones form fluid gums, but the resins are acidic, not aldehydic compounds. Small proportions of easily oxidisable hydrocarbons, such as diolefines, particularly if they are conjugated, promote the formation of much gum. Ordinary olefines give gum more slowly. Traces of acidity cause the discoloration of petrols and paraffins, and are due to sulphur dioxide, its oxidation to sulphuric acid, and the oxidation of mercaptans and alkyl disulphides. Alkalis, oil-soluble bases, and steam, owing to its solvent action, may prevent this type of discoloration. R. A. A. Taylor.

Patents.

Manufacturing active carbon. Naamlooze Vennootschap Algemeene Norit Maatschappij, Assees. of J. N. A. Sauer (E.P. 242,659, 6.11.25. Conv., 8.11.24).—Carbonaceous material in a finely divided state is distilled in vertical, horizontal, or inclined retorts in thin layers. The activating gas is led through ports in the retort wall at right angles to the fuel column, and then parallel with the retort axis to the outlet at the end. The gases flow either in the same direction as the charge or in a reverse direction; the gases evolved, together with the activating gases, are used for heating the retort either internally or externally, and for the preliminary carbonisation of the charge. In one modification the material and gases pass through grates at the end of the retort, and after activation the gases and carbon are withdrawn separately. Several applications of the above principles are described in detail.

A. C. Monkhouse.

Manufacture of charcoal. E. Urbain ([A] E.P. 249,138, 11.3.26. Conv., 12.3.25. [B] E.P. 254,262, 12.3.26. Conv., 25.6.25. [C] E.P. 255,816, 12.3.26. Conv., 22.7.25. Cf. F.P. 606,941, 7.3.25).—(A) Vegetable materials, *e.g.*, peat, wood, straw, etc., are mixed intimately with dicalcium hydrogen phosphate, and then, if necessary, with sulphuric acid, and the mixture, agglomerated and granulated if required, is calcined to give an activated charcoal containing tricalcium phosphate suitable for use in sugar refineries etc. The charcoal can be used for preparing "black paste" (by washing with hydrochloric acid and water successively) or finely divided decolorising blacks. (B) A modification of the process consists in replacing the dicalcium phosphate by a mixture of natural tricalcium phosphate and phosphoric acid. The evolved gases may advantageously be treated to recover the phosphorus as such or as phosphoric acid. (C) Bones are used as starting material in the process above described, the tricalcium phosphate present being converted into dicalcium phosphate by addition of a suitable acid, and any soluble salts then removed by washing. A. B. Manning.

Manufacture of briquettes. S. R. Illingworth, and Illingworth Carbonization Co. (E.P. 258,327, 15.6.25).—Coal is briquetted in moulds arranged on the outside of a drum which is mounted horizontally upon rollers in a casing. The drum is heated internally by gas burners in tubes passing through one end of the casing. The waste gases pass to a return tube in the inside of the drum, and thence to the space between the casing and the insulating jacket. The moulds are fed from a hopper, and a cover plate covers the mould during the preliminary carbonisation. As the drum rotates the briquettes are coked and discharged into an inclined cooling chamber with a discharge valve at the end. The

distillation gases pass into a space above the cover plates and are withdrawn from the apparatus.
A. C. MONKHOUSE.

Regenerative coke ovens. C. A. BAGLIN (E.P. 259,078, 7.1.26).—The air and gas for the first stage of combustion in a vertical-flued oven, of the type having U-shaped heating flues, are admitted at a point above the sole level equivalent to $\frac{1}{2}$—$\frac{3}{4}$ of the width of the oven chamber according to the height of the oven. The gases pass up to the flue, and are met by a second supply of gas admitted in the centre of the two flues, and a further combustion takes place down the second flue. On reversal, the secondary gas supply is automatically drawn into the descending flue. The products of combustion pass through chambers below the sole on their way to the regenerators. A. C. MONKHOUSE.

Fuel for internal-combustion engines. J. D. RIEDEL A.-G. (E.P. 246,182, 19.1.26. Conv., 19.1.25).—Tetrahydronaphthalene or naphthalene is heated above 400° in an atmosphere of hydrogen in the presence of alumina or mixtures of alumina and other metallic oxides; the "cracked" product so obtained is distilled and the fraction below 200° used as a motor fuel, either alone or mixed with alcohol or benzene.
A. C. MONKHOUSE.

Fuel for use in internal-combustion engines. ASIATIC PETROLEUM CO., LTD., and J. KEWLEY (E.P. 259,314, 10.7.25).—By the introduction of 0·33% by weight of nickel carbonyl to kerosene or certain types of gasoline, such mixture affords a fuel which reduces the tendency to "pink" in internal-combustion engines, and increases the critical compression of the fuel. It is particularly applicable in the case of a low-compression fuel. F. G. CROSSE.

Electrical treatment of air [for combustion of oil fuels etc.]. D. F. SARGENT, A. A. LEGAT, and G. R. WITTLETON (E.P. 257,694, 17.6.25).—The conversion of air into oxides of nitrogen is effected by passing it through a chamber consisting of a series of copper electrodes in the form of plates and connecting them to the poles of any suitable source of high-tension unidirectional current, so that there is a continuous, sparkless discharge between them. The gaseous product, when mixed with vaporised oil or petrol fuel, causes considerable economy in the combustion of the fuel in furnaces, ovens, retorts, or internal-combustion engines.
L. M. CLARK.

Gas purifier. J. A. CORNELIER (U.S.P. 1,595,711, 10.8.26. Appl., 21.1.26).—A device for attachment to the exhaust of internal-combustion engines is described.
E. S. KREIS.

Sawdust-distilling apparatus. W. LEE (U.S.P. 1,598,290, 31.8.26. Appl., 4.4.25).—Sawdust is distilled in a retort and the gaseous products are freed from fine particles by means of a deflector and vibrating screen, thus preventing the clogging of the retort connexions.
A. C. MONKHOUSE.

Recovery of cyanide [from ammoniacal gas liquor]. LE R. W. HEFFNER and W. TIDDY, Assrs. to RAINEY-WOOD PROCESS CORP. (U.S.P. 1,600,228, 21.9.26. Appl., 3.3.26).—The gas from an ammonia still is washed with aqueous caustic soda, and the sodium cyanide thus obtained in solution is converted into Prussian blue. The process can be combined with that for the recovery of phenols from ammoniacal gas liquor (cf. U.S.P. 1,566,795—6; B., 1926, 147). The liquor containing sodium phenoxides and cyanide is treated with ferrous sulphate, acidified to separate phenols, and then mixed with ferric chloride to precipitate Prussian blue. T. S. WHEELER.

Apparatus for hydrogenating [and cracking] oils. E. L. ANDERSON (U.S.P. 1,599,629, 14.9.26. Appl., 26.3.23).—An apparatus for cracking oils and hydrogenating the products consists of a cylindrical still surmounted by a column, and fitted with a number of stirrers, each formed of coils of nichrome wire. The column contains a similar coil. A mixture of heavy oil and water is introduced into the still, and maintained at 315—425° by a current passed through the heating coils, which rotate partly in the liquid and partly in the space above. The action of heat, and of currents passing from one coil to another, decomposes the water present, and the hydrogen produced combines with the unsaturated compounds formed by the oil as it cracks. The nichrome acts as a catalyst in these reactions. The oxygen from the water combines with the carbon separated from the oil. The volatile substances thus produced pass up the column, where the various reactions are completed at a temperature of 205—315°. The hydrogen necessary to saturate the products of the cracking can also be supplied by introducing natural gas. A high yield of low-boiling saturated oils is obtained, and there is no deposition of carbon. T. S. WHEELER.

Reclaiming used lubricating oil. AKTIEBOLAGET SEPARATOR, Assees. of P. F. MILLER (E.P. 239,506, 25.8.25. Conv., 6.9.24).—Used lubricating oil is first strained, heated to 71°, and then centrifuged, thus removing heavy solid dirt and water. A counter colloid solution, *e.g.*, sodium phosphate, is added in the proportion of 10 lb. of commercial phosphate and 5 gal. of hot water to 50 gal. of dirty oil. Mixtures of oleic acid and soda ash or soap solutions may also be used. The mixture passes to a centrifuge or gravity settling tank, where the carbon and water are separated The lighter hydrocarbons are then removed in an evaporator by means of a hot air stream from a blower, leaving an oil ready for re-use. A. C. MONKHOUSE.

Manufacture of gasoline. F. M. ROGERS and M. G. PAULUS, Assrs. to STANDARD OIL CO. (U.S.P. 1,599,100, 7.9.26. Appl., 21.7.19).—Oil is distilled pyrogenetically, the distillate is condensed under pressure, and the gas is released by gradual reduction of pressure. W. N. HOYTE.

Treating [cracking] oils. G. KOLSKY (U.S.P. 1,598,973, 7.9.26. Appl., 27.11.25).—High-boiling oils are heated with a mixture of ammonium chloride, or other ammonium salt, and iron, zinc, copper, or aluminium powder under pressure at 150—430°. The ammonium salt and metal react to yield hydrogen and ammonia, and the nascent hydrogen combines with the substances formed by cracking the oil. These low-boiling hydrogenated products are withdrawn at the required pressure with the ammonia from the still. A

good yield of light oils, free from olefines, is obtained, even from heavy oils which have not hitherto been considered suitable for cracking. There is little formation of gas or carbon. T. S. Wheeler.

Cracking of [petroleum] oils. G. E. Heyl (E.P. 259,493, 6.5.26).—Oil is pumped into a removable trough or liner placed in a horizontal retort heated to the desired temperature. The oil is completely converted into vapour and solid carbonaceous residue. The vapours pass through a carbon separator to a dephlegmator. The run-back from the dephlegmator passes to another similar cracking still heated to a somewhat higher temperature, where further cracking takes place. As many stills as desired are placed in series, the dephlegmator run-back passing progressively through the series. The pressure throughout is maintained approximately atmospheric. The removable troughs facilitate the cleaning of the plant. W. N. Hoyte.

Cracking of [hydrocarbon] oils. C. M. Page, Assr. to G. Fabyan (U.S.P. 1,598,618, 7.9.26. Appl., 22.3.22).—In order to heat the vapour space of a cracking still two walls are provided constituting a passage with a constriction. One of the walls at a point adjacent to the constriction is at a high temperature.

W. N. Hoyte.

Cracking of [hydrocarbon] oils. L. B. Cuddy (U.S.P. 1,598,805, 7.9.26. Appl., 10.11.19).—A vertical cracking still is partially enclosed in a furnace; direct contact between the still and furnace gases is avoided by means of baffle walls surrounding the still.

W. N. Hoyte.

Treating hydrocarbon oils. C. P. Dubbs, Assr. to Universal Oil Products Co. (U.S.P. 1,600,721, 7.9.26. Appl., 12.1.20).—The oil to be cracked passes up narrow vertical tubes where it is raised to cracking temperature, and passes thence to an expansion chamber. The vapours evolved pass up a dephlegmator against a downward stream of fresh oil, and thence to a condenser. The mixture of fresh oil and dephlegmator condensate is charged to the lower end of the vertical tubes. Pressure is maintained on the plant as desired. W. N. Hoyte.

Retort for treating oil shales. E. B. Roth (U.S.P. 1,598,882, 7.9.26. Appl., 9.3.25).—Shale is fed on to an inclined disc, rotating within a retort. The disc has baffles built normally from its surface, which have pockets in their sides towards the axis of the disc. The shale is carried in the pockets of the disc through an angle of rotation of 180°, and falls down the inclined face of the disc into the discharging chamber.

S. Pexton.

Manufacture of petrolatum and petrolatum oils. F. Lavirotte (E.P. 259,442, 25.8.25. Addn. to E.P. 240,803, B., 1926, 40).—The first method comprises treatment for 24 hrs. of a mixture of equal parts of crude stock (such as black oils) and refined petrolatum with 20% oleum, using one part of oil to 1·5 parts of acid, the temperature being maintained between 120° and 160°, heat being applied cautiously. When no more sulphur dioxide is evolved the action is considered complete. The liquid is drawn off, neutralised, and freed from suspended carbonaceous matter by filtration through clay in a steam-jacketed filter. The solid resulting from the treatment is broken up and extracted with a suitable solvent, the petrolatum thus obtained being neutralised and filtered as above. A second method comprises treatment of crude stock with 1·5 times its weight of 20% oleum, the acid being slowly added throughout 24 hrs. and the temperature maintained at about 70°. The resulting uniform reddish mixture is heated at 160° for 48 hrs. On cooling to 100° the liquid is drawn off and again sulphonated, together with the petrolatum obtained by extraction of the carbonaceous matter with a solvent, 30% of 20% oleum being used at 150° for 36 hrs. The petrolatum is then neutralised and freed from suspended carbon by filtration as before. The resulting carbonaceous material after extraction is converted into activated charcoal by roasting at 400°. W. N. Hoyte.

Separation of petroleum sludge. I. Hechenbleikner and T. C. Oliver (U.S.P. 1,599,360, 7.9.26. Appl., 16.3.23).—The sludge is separated into its acid and hydrocarbon constituents by heating it under pressure in a lead vessel lined with refractory masonry.

W. N. Hoyte.

Production of purified asphaltic residue from crude petroleum. S. W. Moss, Assr. to The De Laval Separator Co. (U.S.P. 1,599,369, 7.9.26. Appl., 29.9.22).—The oil is heated to a distilling temperature in two stages; between the stages it is centrifuged, thereby cleaning it from solid impurities. An asphaltic derivative containing less than 2% of insoluble matter is produced. W. N. Hoyte.

Manufacture of lubricants [for hot bearings]. M. C. Van Gundy and J. R. Scanlin, Assrs. to Texas Co. (U.S.P. 1,599,854, 14.9.26. Appl., 21.9.23).—Flaked caustic soda (432 pts.) is dissolved in water, and boiled with hard tallow (2680 pts.) until saponification is complete and most of the water has evaporated. A viscous hydrocarbon oil, *e.g.*, steam-refined cylinder stock (2888 pts.), is added, and the mixture is heated at 260° until the water content is less than 1%. The product has m.p. about 230°. It is suitable for use as a lubricant for high-speed bearings running at a high temperature, *e.g.*, locomotive driving journals.

T. S. Wheeler.

Recovering light oils from residual products. A. Oberle, Assr. to T. E. Schofield (U.S.P. 1,599,429, 14.9.26. Appl., 14.4.24).—Vapours of light oils distilled from heavy oils are passed through absorbent activated petroleum carbon and condensed. C. A. King.

Producing lubricating oils by distillation. Simplex Refining Co., Assees. of G. A. Kramer and G. H. van Senden (E.P. 249,801, 17.6.25. Conv., 30.3.25).—Petroleum oil from which the kerosene, gasoline, or other light fractions have been removed, is fed through a tubular still heated to below the decomposition point of the oil into an evaporator containing shallow trays, operated at a pressure of 1 mm. of mercury. The vapours pass through a dephlegmator to a tube condenser, where the lubricating oil is separated. A further condenser, operated at a pressure of 60 mm. of mercury and containing a water spray, is used to

condense the steam. The oil from the evaporator flows to a reheating still, and the vapours distilled are returned to the evaporator. Steam can be admitted either at the entry of the tubular still or to the evaporator.
A. C. Monkhouse.

Motor fuel and process for producing same. J. M. Hirsh (E.P. 258,913, 5.5.25).—Air and steam are passed through a generator containing red-hot carbonaceous material; the carbon monoxide, hydrogen, and undecomposed steam produced are led through a bed of tin plate or other metallic scraps resting on a grate above the fuel. The gases then pass through the tubes of a steam boiler to closed coils in a still containing crude petroleum or heavy paraffin oils. The distillate is collected in a tank, where it is admixed with the cooled gases under pressure, yielding a motor fuel.
A. C. Monkhouse.

Purification of crude alcohols [from oil gas]. R. de M. Taveau, Assr. to Texas Co. (U.S.P. 1,600,437, 21.9.26. Appl., 5.8.24).—*iso*Propyl alcohol from cracked petroleum gases is purified by distillation over 1—2% of solid sodium hydroxide. T. S. Wheeler.

Apparatus for quenching and conveying discharged coke. A. Barnes, R. W. Broadhead, and R. Dempster & Sons, Ltd. (E.P. 259,325, 18.7.25 and 24.8.25).

Treatment of gas liquors for removing phenolic impurities therefrom. Le R. W. Heffner and W. Tiddy (E.P. 244,775, 15.12.25. Conv., 16.12.24).—See U.S.P. 1,566,795—6; B., 1926, 147.

Removing hydrogen sulphide from gases. T. P. L. Petit (U.S.P. 1,598,985, 7.9.26. Appl., 7.3.23. Conv., 16.3.22).—See E.P. 195,061; B., 1923, 929 A.

Distilling oil. L. E. Hirt (E.P. 258,436, 12.10.25).—See U.S.P. 1,559,701; B., 1926, 40.

Process for decolorising and stabilising oils. P. W. Prutzman, Assr. to General Petroleum Corp. (Reissue 16,439, 12.10.26, of U.S.P. 1,547,682, 28.7.25).—See B., 1925, 749.

Purification systems for the lubricating oil of internal-combustion engines. Sharples Specialty Co., Assees. of P. T. Sharples and L. D. Jones (E.P. 231,877, 2.4.25. Conv., 2.4.24).

Liquid fuel burners. A. Lang (E.P. 237,587, 6.7.25. Conv., 28.7.24).

Burner for heavy oil. E. Sambuc and L. Brazzola (E.P. 252,400, 25.5.26).

Gasification of heavy hydrocarbons [for internal-combustion engines]. L. Mirlesse (E.P. 250,220, 24.3.26. Conv., 3.4.25).

Cracking hydrocarbon oils. W. F. Faragher, W. A. Gruse, and F. H. Garner, Assrs. to Gulf Refining Co. (U.S.P. 1,601,727—8, 5.10.26. Appl., 15.1.21).—See E.P. 174,087—8; B., 1923, 301 A.

Furnaces (E.P. 259,389).—See I.

Desulphurising naphthalene or paraffin (E.P. 260,129).—See III.

III.—TAR AND TAR PRODUCTS.

History and composition of low-temperature tar. E. Parrish (Fuel, 1926, 5, 436—465).—The results of researches on the composition of low-temperature tars are collated and examined in detail. Great variation is found in the content of the neutral, acidic, and basic compounds of the tars and their behaviour on distillation. Low-temperature tars contain "ulmins" insoluble in common organic solvents, but soluble in pyridine or phenols; neutral oils, comprising 50—80% of the tar, and consisting of paraffins, naphthenes, aromatic and unsaturated hydrocarbons, together with oxygen and sulphur compounds; acidic compounds, viz., phenols and carboxylic acids, the phenols, forming 10—50% of the tar, being mainly cresols, xylenols, and higher homologues of phenol, together with high-boiling resinous phenols; and basic compounds which are homologues of pyridine and quinoline and hydrogenated derivatives, together with high-boiling viscous bases. The yield of low-temperature tar is greater from "young" coals and coals rich in oxygen. A. C. Monkhouse.

Basic compounds in low-temperature tar. Y. Oshima and K. Ishibashi (J. Soc. Chem. Ind. Japan, 1926, 29, 445—451).—The tar examined contained 0·67% of basic substance, of d^{15} 0·984. The greater part distils at 100—300°, and about 60% above 200°. 2-Methylpyridine, 3 : 4-dimethylpyridine, and 2 : 4 : 6-trimethylpyridine were isolated. The presence of aniline and its homologues was also confirmed. K. Kashima.

Occurrence of thiophen, benzene, and their next higher homologues, in tar oil from Russian bituminous schists. J. Dodonov and E. Sochestvenskaja (Ber., 1926, 59, 2202—2208).—The isolation of thiophen and its homologues is possible only after unusually thorough fractional distillation of the crude oil. Benzene, toluene, and thiophen are thus detected. After treatment with dilute sulphuric acid, soda-lime, sodium, and ammonia, the presence of 2-methylthiophen and 2 : 3-dimethylthiophen is established in certain fractions. H. Wren.

Patents.

Distillation or cracking of tars, petroleum oils, and similar materials. C. R. Downs (E.P. 258,433, 7.10.25).—To prevent the decomposition of vapours in the distillation of tars etc., the material to be distilled is heated externally in coils of cast iron or calorised steel by means of sulphur vapour under pressure. Temperatures of 215—400° (the decomposition point of coal tar) are obtained by adjustment of the pressure with the aid of a neutral gas. Owing to the low density of sulphur only a small sulphur boiler is required.
A. C. Monkhouse

Continuous distillation of tar and like substances and continuous rectifying of their by-products. C. Ab-der-Halden (E.P. 253,935, 18.6.26. Conv., 22.6.25. Addn. to E.P. 239,841; cf. B., 1915, 982).—The process described in the main patent is modified by extending the pipe through which the hot raw material is supplied below the surface of the mass of residue in the distilling retort. The water contained in the raw material is vaporised in the retort, and thus aids the distillation process. A. B. Manning.

Desulphurising naphthalene or paraffin. K. BUBE (E.P. 260,129, 22.12.25).—The solid material is pugged with comparatively small amounts of alcohol or acetone, with or without the addition of benzene or benzine, and the solvent then separated. The sulphur compounds are removed with inappreciable loss of the hydrocarbon itself. A. B. MANNING.

IV.—DYESTUFFS AND INTERMEDIATES.

PATENTS.

Purification and isolation of anthraquinone-β-sulphonic acid. NEWPORT Co. (E.P. 251,974, 28.4.26. Conv., 9.5.25).—Anthraquinone-β-sulphonic acid is isolated as its ammonium salt. For example, the sulphonation mass obtained by heating 1 pt. of anthraquinone with 1 pt. of 40% oleum at 145° for 2 hrs. is poured into 10 pts. of cold water. Unchanged anthraquinone is filtered off and the filter cake washed with warm water. The combined filtrates are concentrated to d^{25} 1·07 and concentrated aqueous ammonia is added until slightly alkaline. The solution is heated to 90°, kept at this point for 1 hr., and cooled slowly to 25°. After 12 hrs. the crystals of ammonium salt are filtered off and dried at 100°. A. DAVIDSON.

Preparations of [vat] dyestuffs. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (E.P. 257,991, 8.6.25).—Vat dyes are mixed, and in some cases milled, with sulphite-cellulose waste liquor or a salt of a ligninsulphonic acid together with, in some cases, a salt of a sulphonated soap, and the pastes so obtained are dried. Such preparations give colloidal suspensions in water which are very easily vatted, and are particularly suitable for use in cotton printing. In the case of indigoid dyes the products obtained do not require a caustic alkali for vatting, but may be vatted with sodium carbonate and hyposulphite, thus facilitating the application of such dyes to wool. A. DAVIDSON.

Halogenated indigoid dyestuffs. H. STAUDINGER, R. TOBLER, R. STOCKER, J. MÜLLER, and A. BUCHER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,600,743, 21.9.26. Appl., 28.10.25).—See E.P. 236,332; B., 1925, 703.

Azo dyestuffs and process of making same. M. ISLER and L. VON MECHEL, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,600,763, 21.9.26. Appl., 9.12.24. Conv., 28.12.23).—See E.P. 226,797; B., 1925, 703.

Preparation of derivatives of naphthoquinone. A. WAHL and R. LANTZ, Assrs. to SOC. ANON DES MAT. COL. ET PROD. CHIM. DE SAINT-DENIS (U.S.P. 1,599,444, 14.9.26. Appl., 21.12.22. Conv., 28.12.21).—See E.P. 191,064; B., 1923, 1044 A.

Production of tetrazoles. K. F. SCHMIDT, Assr. to KNOLL & Co. (U.S.P. 1,599,493, 14.9.26. Appl., 11.12.25. Conv., 2.1.24).—See E.P. 257,418; B., 1926, 932.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of concentrated sodium hydroxide solutions on flax yarn and fabrics under conditions of mercerising. P. P. VIKTOROV [with N. N. MALYUTIN] (J. Text. Ind. Com. [Moscow], 1924).—The elasticity of flax fabric increases under the action of sodium hydroxide solutions, but not so much as cotton fabric. The absolute tensile strength of flax fabric decreased to 93%, and of cotton fabric increased to 125% of its initial strength under the action of sodium hydroxide (d 1·265). A. A. ELDRIDGE.

Swelling of sulphite-cellulose in sodium and potassium hydroxides at different temperatures. O. FAUST (Cellulosechem., 1926, 7, 153—155; cf. B., 1925, 311).—A technical method for the comparison of the swelling action of various alkaline solutions on cellulose is described. The cellulose (1 g.) in paper form is immersed in 100 parts of the solution at a constant temperature for 1½ hours, after which it is allowed to drain to constant weight. The increase in weight is taken as a measure of the degree of swelling, although the amount of liquor retained by the fibre under these conditions is also dependent to some extent on the surface tension and viscosity of the solution. According to the results of this method, sodium hydroxide at 0° and at 20° has a much greater effect on sulphite-cellulose than has potassium hydroxide, and is most effective at a concentration of 10% (by weight) at both temperatures. A solution of this concentration also has the greatest solvent action on sulphite-cellulose (Bubeck, B., 1926, 579). The swelling action of potassium hydroxide at 0° increases with increasing concentration until a 16·8% solution is reached, after which it remains approximately constant. At 20° there appear to be two stages in the process. The degree of swelling increases as the concentration of potassium hydroxide rises to 5·6%, remains constant between 5·6 and 14%, then increases again to a maximum at a concentration of 28%. The viscosities of both sodium and potassium hydroxide solutions increase continuously with increasing concentration, and it is therefore unlikely that the results are appreciably influenced by this factor. W. J. POWELL.

Swelling of cotton cellulose in sodium and potassium hydroxides of different concentrations. O. FAUST (Cellulosechem., 1926, 7, 155—156; cf. B., 1925, 984).—The method outlined in the preceding abstract for the comparison of the degree of swelling of cellulose in various alkaline media is applied to cotton cellulose. With sodium hydroxide at 20°, the degree of swelling–concentration curve exhibits a maximum at 10% of sodium hydroxide (by weight). The peak is sharper than in the corresponding curve for sulphite-cellulose, due possibly to the fact that the latter substance contains a larger proportion of alkali-soluble material. In solutions of potassium hydroxide below 40% the results for cotton cellulose are similar to those obtained with sulphite-cellulose. Solutions of more than 40% of potassium hydroxide cause a sharp increase in the degree of swelling of cotton cellulose, the fibre is considerably shortened, and suffers a 50% reduction in surface area. The swelling in 10% sodium hydroxide solution is a different phenomenon, as little reduction in the surface area of the fibre is apparent. W. J. POWELL.

Soda pulp investigations. I. Yield and quality of pulp as affected by length of chip. D. E. CABLE, R. H. MCKEE, and R. H. SIMMONS (Pulp and Paper Mag., 1926, **24**, 1249—1252).—Experiments have been made

to determine whether any tendency towards selective cooking, arising from the different permeabilities of aspen and the hard woods, could be corrected by suitably adjusting the length of chip. In no case, however, was it found that, between the limits 0·5 and 1·25 in. (with the grain), the size of the chips had any appreciable influence on the yield and bleach consumption of the pulp. The average yields of soda pulp under standard conditions for aspen, white birch, and white maple were respectively, 48·2, 46·4, and 45% (bone-dry pulp on bone-dry chips). Expressed as pounds of air-dry bleached pulp per cord of wood, these figures become 1176, 1895, and 2051 respectively, showing that, owing to their greater density, the hard woods actually give higher yields on the cord basis than does aspen. The bleach requirement of the hard woods proved, however, to be considerably higher, viz., approximately 13% as against 6·3% for aspen cooked under the same conditions.

D. J. NORMAN.

Use of glue in coated paper. G. K. HAMILL, V. H. GOTTSCHALK, and G. W. BICKING (U.S. Bureau Standards, Tech. Paper No. 323, 1926, **20**, 635—666).—Paper was coated by means of an experimental plant with glues of different grades as adhesives, and using casein for comparison, together with the different types of coating minerals, clay, satin white, and blanc fixe. The glues were graded in accordance with their grease content, colour, odour, acidity, adhesiveness, and clay-suspending power. The proportion of glue required in a coating mixture was determined by making experimental coatings with increasing amounts of glue in the mixture, and subjecting the paper to the sealing-wax pick test and also to printing tests. The proportion of glue needed to give a good coating varied inversely with the grade of glue, the amount of the better-grade glues required being often less than that of casein. The glue-coated paper is not so waterproof as that coated with casein, but it may be improved in this respect by hardening agents such as alum or formaldehyde. The use of glue gives a higher finish to the paper, whilst with neutral glues a wider range of colours is available than with the alkaline casein. Little change is needed in equipment and operating technique from that used with casein, whilst glue-coated papers present no difficulty in printing except where a high degree of water-resistance is required. F. R. ENNOS.

Chitin. E. KNECHT and E. HIBBERT (J. Soc. Dyers and Col., 1926, **42**, 343—345).—Dried, powdered crab-shell (ash, 50·5%) after four alternate treatments with hydrochloric acid and sodium hydroxide, extraction with ether, drying, sieving (60 mesh), and final solution in cold concentrated hydrochloric acid and precipitation with water, gives a 25% yield of ash-free chitin, affording 90·4% of glucosamine on boiling with hydrochloric acid. The chitin thus obtained is unattacked by cold nitric acid (d 1·425) and potassium permanganate in 10% sulphuric acid, is insoluble in Schweizer's solution, and does not reduce Fehling's solution. Organic solvents have little or no effect on the purified chitin, and in its stability towards alkalis chitin offers advantages over silk, but is less readily dyed. Crystal Scarlet in the presence of sulphuric acid readily dyes chitin, but Diamine Sky Blue only affords a weak shade, and Methylene Blue does not dye pure chitin. With excess of iodine in potassium iodide chitin becomes almost black, but dries to a light brown. It appears to be closely related to "sea silk" the byssus from *Pinna squamosa*, L. (cf. Kunike, Kunstseide, 1926, 8, 182). R. BRIGHTMAN.

PATENTS.

Manufacture of threads, filaments, and the like from viscose. COURTAULDS, LTD., H. J. HEGAN, and F. BAYLEY (E.P. 259,386, 17.10.25).—When producing hollow filaments from viscose containing sodium carbonate, more satisfactory results are obtained by using a solution containing not more than 6% of cellulose rather than the customary 7%. *Example*: 100 pts. of air-dry cellulose are impregnated with caustic soda (d 1·2), squeezed to 300 pts., and exposed for 3 hrs. at 25° to the action of 30—40 pts. of carbon disulphide. The resulting xanthate is dissolved in dilute caustic soda, and, after the addition of a dilute solution containing 43 pts. of sodium carbonate, the cellulose and caustic soda contents of the mixture are adjusted to 6 and 5% respectively. The viscose solution thus obtained is filtered, freed from air in a vacuum, and projected into a spinning bath. The lustre and degree of inflation of the filaments are modified by the composition of the spinning bath and the ripeness of the viscose; for example, the higher the proportion of zinc sulphate in the spinning bath, and the shorter the time of ripening of the viscose, the greater will be the degree of inflation.

D. J. NORMAN.

Treatment of threads, fabrics, or other materials composed of or containing artificial filaments. BRITISH CELANESE, LTD., J. F. BRIGGS, J. T. KIDD, and C. W. PALMER (E.P. 259,265, 26.5.25).—Cellulose acetate silk, either alone or in admixture with other fibres, may lose its lustre during the various processes to which fabrics are exposed. This may be restored completely or to any desired extent by treating the fabric with aqueous solutions of one or more substances which are solvents or swelling agents for cellulose acetate. Such substances include acetic acid, phenol, benzyl alcohol, triacetin, *cyclo*hexanone, ammonium and other thiocyanates. The duration of the treatment depends on the concentration of the swelling agent, the degree of lustre it is desired to impart, and the temperature, but the conditions must not be such that fusion or permanent deformation of the filaments results; in general, the temperature should not exceed 30—40°, and 1—5 hrs. is a convenient time of exposure. After treatment the fabric is centrifuged and allowed to dry, preferably without rinsing, at the ordinary temperature. Rinsing is avoided in order that the ratio of swelling agent to water in the liquid retained by the filaments should not be materially reduced. For this reason it is better to use those solvents which have a b.p. higher than that of water. The following are examples of suitable solutions and temperatures: 15—20% of acetic acid (by weight) at 20°, 1·5% solution of phenol at 15—20°, a saturated solution of benzyl alcohol at 20—30°, a saturated solution of triacetin (about 1%) at 20—30°, 5—7% solution of *cyclo*hexanone at 20°, or a solution of ammonium

thiocyanate (200—250 g./litre) at the ordinary temperature. D. J. Norman.

Protection of cotton and other textile or like materials from mildew. British Dyestuffs Corp., A. Renshaw, and T. H. Fairbrother (E.P. 259,690, 24.7.25).—The development of mildew on cotton and other textile materials, including paper and leather, is prevented by impregnating the material with a 0·1—1% solution of an alkali salt of a halogenated phenol containing more than two atoms of halogen in the molecule, *e.g.*, trichloro- or tribromo-phenol. The halogenated phenol compound may be applied by spraying, or may be added to the dye bath, or, in the case of artificial silk, to the spinning solution, or, in the case of paper, to the beaten pulp or the size. Paper thus treated is particularly suitable for wallpaper, legal documents, tubes, and in connexion with the storage and transportation of foodstuffs. D. J. Norman.

Treating textile fibres, such as esparto grass, alfa, and similar vegetable substances. Soc. Anon. A.L.F.A. Applicazioni Lavorazioni Fibra Alfa e affini (E.P. 256,570 and 259,452, 25.2.26. Conv., 5.8.25 and 12.11.25).—(A) Long straight flexible fibres, suitable for cordage or for making sacks, are produced from esparto and the like by digesting the raw material with an alkaline solution for about 8 hrs. at 98—180°, washing, drying, and passing the dry leaves between rollers to open up the fibres and remove impurities. The final separation of the fibres may be effected by hackling machines. (B) A modification of the above process is described in which the digested and dried leaves are arranged parallel to each other, and rolled in the direction of hackling. Means are preferably provided for bending the leaves in both directions during rolling and for increasing the pressure on the rollers towards the end of the operation. Suitable conditions of digestion are: 8 hrs. at a pressure of 3 atm. with a solution containing 3% (on the weight of grass) each of Solvay soda (*d* 2·1) and sodium silicate. After digestion the fibres may be further softened by washing with an oil emulsion containing, for example, 2 litres of olive oil and 100 g. of Solvay soda per 400 litres of water. D. J. Norman.

Manufacture of artificial silk [cuprammonium cellulose]. Brysilka, Ltd., and F. W. Schubert (E.P. 258,371—6, 2.7.25).—(A) The crude filtered viscous solution is fed by a pump through tubes, surrounded by a cooling medium, to a mixing chamber constituted by rotary blades or stirrers bearing against the outlet ends of the tubes from which the solution is issuing. (B) In the stretch-spinning process of cuprammonium solutions of cellulose the coagulating liquid is fed into the spinning funnel or cylinder from below under atmospheric pressure. The upper portion of the funnel, where the head carrying the spinning nozzle enters the liquid, is closed, while the lower open end is suspended below the surface of the liquid in a tank, but a suction orifice is provided at the top through which the air can be exhausted from the funnel when the operation is being started up. Circulation and replenishment of the liquid in the cylinder are effected by the surface friction of the threads passing downwards through the column of liquid in the tube. (C) The cuprammonium solution is fed under pressure to a measuring pump comprising a casing in which is a cylindrical rotary member containing radially disposed recesses and sliding pistons, the latter being actuated by projecting pins and runners which engage in eccentric grooves formed in the side cover-plates. The quantity of fluid discharged by the pump is finely adjusted by mounting the side cover-plates in flanged rings, in which they can be rotated through a small angle so as to vary the position of maximum throw of the eccentric grooves in relation to the outlet. The feed inlet may also be controlled by a screw-adjusted plunger with conical end. (D) In fixing the spinning cylinder or funnel to the spinning head, the spinneret-top is secured to a ring by means of a locking plate rotatably mounted on the spinneret-top and adapted to engage projections on the ring forming a bayonet joint. By slight rotation of the locking plate, rubber or other packings are compressed between the ring and the spinning cylinder, and between the spinneret-top and the ring. (E) The coagulated thread is washed with water, acid, or other liquid, by conducting it over a tray attached to the edge of the tank, having transverse ridges on its floor, and inclined so that the thread travels upwards over the ridges while liquid flows downwards in counter-current. The tray is constructed in two portions, which may be connected by a flexible tube for single washing or to two independent liquor systems for double treatment. The front portion of the tray is triangular, the fresh liquid being introduced at the apex, which terminates in the form of a thread-guide. Drainage holes are provided at the bottom of both portions of the tray, and means are provided for reciprocating the front triangular portion to traverse the thread on the winding bobbin. (F) The winding bobbin consists of a hollow cylindrical shell frictionally supported on a frame or spider. The latter consists of two three-armed end pieces with slotted connecting bars between, the slots being fitted with rubber-covered spiral springs. The springs normally are slightly too long for the slots, and have an outward thrust against the periphery of the shell, but are capable of compression into the slots when the shells are being changed.
J. F. Briggs.

Process for producing chemical wood pulp. G. A. Richter, Assr. to Brown Co. (U.S.P. 1,598,880, 7.9.26. Appl., 12.7.26).—Raw cellulosic material is heated with the spent liquor resulting from the alkaline digestion of unbleached cellulosic pulp, made acid with sulphur dioxide. S. Pexton.

Reducing the viscosity characteristics of nitrocellulose materials. V. E. Kimmel, Assr. to Eastman Kodak Co. (U.S.P. 1,598,972, 7.9.26. Appl., 25.2.25).—Nitrocellulose material, *e.g.*, film scrap free from gelatin, is immersed in a saturated solution of calcium hypochlorite for 1—7 days, and yields solutions of reduced viscosity in the usual solvents, which can be applied to the manufacture of lacquers for spraying purposes.
T. S. Wheeler.

Manufacture of new cellulose derivatives. L. Lilienfeld (E.P. 252,654, 16.7.25. Conv., 30.5.25).—Derivatives of cellulose xanthic acids, which from their

mode of formation and chemical characteristics are probably esters of cellulose xanthic acid of the type $C_{6n}H_{10n-1}O_{5n-1})\cdot O\cdot CS\cdot S\cdot R$, where R indicates an alcohol radical, are obtained by acting on cellulose xanthic acid or a cellulose xanthate with an inorganic acid ester under faintly alkaline, neutral, or acid conditions. The parent material may be either crude or purified viscose, or a heavy metal (zinc) salt of cellulose xanthic acid, provided that the free alkali present is less than 20%, preferably less than 10%, calculated as sodium hydroxide on the weight of cellulose. Adjustment of the alkalinity is made with dilute acetic acid. Esterification proceeds without the application of heat, but, if necessary, the reaction may be started or accelerated by warming. *Example*: 1000 pts. of crude viscose containing 100 pts. of parent cellulose are diluted, either in the fresh state or after keeping for 6 hrs. to 3 days, with 9000—10,000 pts. of water, and treated while stirring with 5—10% acetic acid until the solution contains free alkali within the limits specified above, or is neutral, or acid. When the hydrogen sulphide resulting from the neutralisation has to a large extent escaped, 100 pts. of ethyl sulphate are added and the mixture is stirred. After about an hour the mass becomes gelatinous, but later liquefies and gives a fine flaky precipitate. This precipitate is collected, either immediately or after keeping for several hours or days, washed, and dried. The product is, after comminution, a white powder soluble in 8—10% caustic soda solution and in many organic solvents, *e.g.*, aqueous 70—80% pyridine solution. These esters of cellulose xanthic acid may be used for the manufacture of transparent flexible films, filaments, plastic masses, etc., for which purpose they are dissolved in 8—10% alkali solution and coagulated.

D. J. NORMAN.

Cellulose-ester plastic and solution. I. G. FARBENIND. A.-G., Assees. of O. SCHMIDT, T. EICHLER, and K. SEYDEL (U.S.P. 1,600,700, 21.9.26. Appl., 26.2.26. Conv., 27.12.22).—Esters of hydroaromatic alcohols with an aliphatic dicarboxylic acid, excepting adipic acid, are valuable plasticisers and solvents for cellulose nitrate and acetate. T. S. WHEELER.

Cellulose ether solvent and composition. L. LILIENFELD (U.S.P. 1,599,569, 14.9.26. Appl., 8.1.23).—The solvent consists of a mixture of nitromethane and a lower monohydric aliphatic alcohol. D. J. NORMAN.

Manufacture of high α-cellulose fibre. G. A. RICHTER and M. O. SCHUR, Assrs. to BROWN Co. (U.S.P. 1,599,489, 14.9.26. Appl., 9.4.25).—Unbleached pulp is treated with an oxidising liquor and then digested with a solution of lime. D. J. NORMAN.

Preparing paper half-stock. A. MACKAY (U.S.P. 1,599,831, 14.9.26. Appl., 10.1.22).—Two batches of cellulose pulp are chemically hydrated, one more strongly than the other, and are beaten separately during the hydration, the resulting stocks being subsequently mixed in any desired proportion. D. J. NORMAN.

Apparatus for washing wool etc. Process and apparatus for washing or otherwise treating wool etc. E. C. DUHAMEL, and COMP. GÉN. DES INDUSTRIES TEXTILES (E.P. 234,488 and 258,895, 20.5.25. Conv., 0.5.24).

Manufacture of fibrous materials. RADUNER & Co., A.-G. (E.P. 251,993, 4.5.26. Conv., 11.5.25).—See G.P. 423,858; B., 1926, 403.

Manufacture of moulded or pressed goods from fibrous materials. F. KAYE (U.S.P. 1,600,047, 14.9.26. Appl., 4.9.23. Conv., 7.11.22).—See E.P. 210,193; B., 1924, 250.

Manufacture of threads, filaments, strips, or films from cellulose compounds. H. J. HEGAN, Assr. to COURTAULDS, LTD. (U.S.P. 1,599,233, 7.9.26. Appl., 13.1.25. Conv., 2.2.24).—See E.P. 229,075; B., 1925, 312.

Manufacture of threads, filaments, strips, or films from cellulose ethers. W. H. GLOVER, Assr. to COURTAULDS, LTD. (U.S.P. 1,599,230, 7.9.26. Appl., 17.11.24. Conv., 5.1.24).—See E.P. 224,405; B., 1925, 38.

Making cellulose acetate directly spinnable from reaction mixtures in commercial form. J. O. ZDANOWICH (U.S.P. 1,600,159, 14.9.26. Appl., 24.8.25. Conv., 15.8.24).—See E.P. 244,148; B., 1926, 152.

Purification of cellulose ethers. J. ALTWEGG and C. A. MAILLARD, Assrs. to SOC. CHIM. USINES DU RHÔNE (U.S.P. 1,599,508, 14.9.26. Appl., 20.10.25. Conv., 22.12.24).—See F.P. 603,791; B., 1926, 739.

Drive for machines for spinning artificial silk. O. VON KOHORN and A. LEHNER (E.P. 250,198, 12.3.26. Conv., 6.4.25).

[Mechanical] treatment of jute, hemp, etc. P. SHARP (E.P. 258,929, 26.6., 16 and 18.9.25).

Drying apparatus for textiles. H. HAAS (E.P. 244,043, 18.7.25. Conv., 4.12.24).

Making sulphite cooking liquors. (U.S.P. 1,599,488).—See VII.

Sulphur dioxide from blow-pit gases. (U.S.P. 1,599,490).—See VII.

Wall coverings [from pulp] (E.P. 259,826).—See IX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Formation of insoluble colours on cellulose acetate silk. J. POKORNÝ (J. Soc. Dyers and Col., 1926, **42**, 345—348).—Cellulose acetate silk absorbs and fixes many intermediate compounds, diazo- and tetrazo-compounds as such, and the process originally described (J.S.C.I., 1894, **13**, 388) for the production of insoluble colours on animal fibres has now been applied to the dyeing of acetate silk. Thus cellulose acetate silk is worked for a few minutes in the fine suspension obtained by pouring into water a hot alcoholic solution of β-naphthol, washed from mechanically adherent naphthol, and passed into a solution of diazotised *p*-nitroaniline. The process may be reversed. Intermediates such as phenylmethylpyrazolone, which are soluble in water, afford much deeper shades if a little alcohol is added to the aqueous solution to produce a fine suspension. Para Red may also be produced on cellulose acetate silk by passing the silk through the mixed suspension of the components in dilute alcohol and then washing and diazotising. Meldola's Blue may be similarly developed on cellulose acetate

silk by the use of a mixed suspension of β-naphthol and *p*-nitrosodimethylaniline and steaming. Toluylene Blue, when similarly produced on cotton, silk, and viscose silk, on further steaming changes into Neutral Red; on cellulose acetate silk the blue does not change either on prolonged steaming or on hot soaping, but alters on keeping (within three years). Aniline Black may be developed on cellulose acetate silk by working the material in the suspension of Diphenyl Black Base I (M.L.B.), washing, and oxidising in a solution of ammonium chloride, sodium chlorate, and a vanadium solution. R. Brightman.

Behaviour of different starches towards dyestuffs. Huebner and Venkataraman.—See XVII.

Patents.

Bleaching [textile materials] with hypochlorites. A. L. Mond. From I. G. Farbenind. A.-G. (E.P. 260,190, 22.6.26).—Cellulosic materials are bleached with hypochlorites in the presence of a metallic catalyst such as salts of cobalt, copper, and especially nickel. The catalyst accelerates the rate of bleaching. The efficiency of a nickel catalyst is favourably influenced by the presence of hydroxyl ions, and coloured nickel compounds, *e.g.*, black nickel oxide, remaining in the bleached material may be easily removed by washing with a dilute solution of an acid or bisulphite. Usually less than 0·5% of a nickel salt (based on the weight of material to be bleached) is satisfactory. A. J. Hall.

Dyeing [cellulosic materials]. J. Y. Johnson. From Badische Anilin u. Soda Fabrik (E.P. 259,634, 15.5.25).—Cellulosic materials chemically combine with derivatives of isatoic anhydride when dyed with it, and the resulting dyed material may be further modified in colour by diazotisation and coupling (when diazotisable amino-groups are present in the isatoic compound), or by coupling with diazotised compounds. Especially suitable isatoic compounds are derived from azo dyes having a carboxyl group with an amino-group in the *ortho*-position, and may be obtained by the action of a carbonic acid halogenide on *o*-aminocarboxylic acids, phosgene and alkyl chloroformates being suitable halogenides. Dyeing is carried out in the manner usually employed for direct dyes, the dye bath being maintained cold and slightly alkaline. The isatoic compound obtained by treatment with phosgene in aqueous alkaline solution of *p*-sulphobenzeneazoanthranilic acid dyes cotton an intense yellow shade, which yields a bright red shade when diazotised and coupled with β-naphthol. Other suitable compounds are prepared from 2-chloro-5-sulphobenzeneazoanthranilic acid, 2 : 3-napthylaminecarboxylic acid, and *p*-nitroanthranilic acid. A. J. Hall.

Dyeing cellulose ethers. H. Eichwede and E. Fischer, Assrs. to Grasselli Chem. Co. (U.S.P. 1,599,748, 14.9.26. Appl., 9.11.25. Conv., 25.11.24).—Cellulose ethers are dyed with a nitroarylazodiethylaniline-*m*-sulphonic acid in which the nitrated aryl nucleus is not sulphonated.

Dyeing machine. W. E. H. Bell, Assr. to H. W. Butterworth & Sons Co. (U.S.P. 1,600,574, 21.9.26. Appl., 20.11.24).

Apparatus for colouring portions of yarn in mass. A. E. White. From Randomtex Dyeing Machine Corp. (E.P. 259,793, 25.11.25).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Production of pure zirconia from semi-refined zirconia ores. K. Tabata and S. Moriyasu (J. Soc. Chem. Ind. Japan, 1926, **29**, 476—481).—The material used contained 10·33% SiO_2, 77·96% ZrO_2, 4·26% Al_2O_3, 1·87% Fe_2O_3, 1·24% TiO_2, 0·57% P_2O_3, and a trace of BeO; ignition loss 3·09%. The powdered and elutriated ore, mixed with about four times its weight of sulphuric acid, was evaporated to dryness, the residue heated at 350—400°, avoiding oxidation of ferric sulphate etc. and expelling free sulphuric acid as completely as possible. A filtered aqueous extract of the dried residue was neutralised with sodium carbonate and kept for about 24 hrs., when a large amount of basic zirconyl sulphate was precipitated. This when washed with alcohol and dried consisted of $8ZrO_2,7SO_3$ containing iron and titanium as impurities. When dissolved in hydrochloric acid (0·5—1*N*) and boiled the zirconium was precipitated as $2ZrO_2,SO_3,5H_2O$. By repeating the process 3 or 4 times the iron and titanium were completely eliminated. K. Kashima.

Dehydration of metallic salt hydrates. I. Dehydration of sodium borate, carbonate, and sulphate. M. A. Rakusin and D. A. Brodski (Z. angew. Chem., 1926, **39**, 1345—1348).—Cold 95% alcohol dehydrates completely the decahydrate of sodium sulphate, has no effect on borax crystals, and removes about 75% of the water from sodium carbonate decahydrate. Sodium sulphate is also completely dehydrated in 48 hrs. by 80% alcohol, and in 9 hrs. in a current of dry air at the ordinary temperature. Sodium carbonate decahydrate is more slowly converted into the monohydrate in dry air at 15°, and on exposure to the atmosphere this compound is converted into the stable dihydrate, which is also the final product obtained by prolonged exposure of the decahydrate to the atmosphere. Borax remains completely unchanged on exposure to damp or dry air for prolonged periods. The reason for the different behaviour of the three decahydrates is to be found in their heats of formation from the anhydrous salt; these are 36·0, 21·8, and 18·82 g.-cal. respectively for the borate, carbonate, and sulphate.
A. R. Powell.

Decomposition of ammonium thiocyanate into carbon disulphide and mellon, and decomposition of mellon into ammonia and carbon dioxide. W. Gluud, K. Keller, and W. Klempt (Z. angew. Chem., 1926, **39**, 1071—1073).—When a 24% solution of ammonium thiocyanate is slowly dropped on to a hot nickel plate in a thermostat maintained at 230—260° complete decomposition of the salt is effected into ammonia, carbon disulphide, and mellon, together with smaller quantities of other nitrogenous substances and a variable proportion of hydrogen sulphide. Under carefully controlled conditions on a semi-large scale 1 kg. of ammonium thiocyanate should yield 362 g. of carbon disulphide, 1188 g. of ammonium sulphate by absorption

of the ammonia in 10% sulphuric acid, and 219 g. of mellon. The last named is a light-brown, granular, amorphous mass, which is completely decomposed into ammonia and carbon dioxide by heating at 500° in an iron tube through which a current of steam is passed: $C_6H_3N_9 + 12H_2O = 6CO_2 + 9NH_3$. In view of this reaction it is suggested that mellon may be of value for direct use as a fertiliser. An apparatus for the continuous decomposition of the thiocyanate solution on a large laboratory scale is described and illustrated.

A. R. Powell.

Complete analysis of fluorspar. E. Bailleux (Bull. Soc. chim. Belg., 1926, **35**, 305—310).—Loss on ignition is determined by heating 2 g. of the sample at 300° until the weight is constant. The residue is digested with 150 c.c. of 10% acetic acid for 30 min. on the water bath, and the insoluble portion is collected, ignited at 300°, and weighed; loss in weight is reckoned as calcium carbonate. Exactly one-half of the residue (= 1 g. of original sample) is evaporated with 3—4 c.c. of concentrated sulphuric acid and 3 c.c. of hydrofluoric acid to expel silica and convert all fluorides into sulphates. The residue is dissolved in 10% hydrochloric acid and the solution used for the determination of iron and alumina and of the lime present as fluoride and sulphate. Silica is determined from the loss of weight when the second portion of the acetic acid-insoluble is evaporated with hydrofluoric acid to dryness and the residue calcined after again evaporating with ammonia. The residue is digested with nitric and hydrofluoric acids, and the excess is expelled on the water bath; 50 c.c. of ammoniacal acetate–citrate mixture (500 c.c. of 65% acetic acid and 80 g. of citric acid made up to 1 litre with ammonia) are added to dissolve calcium sulphate and ferric fluoride, leaving a residue of pure calcium fluoride which is collected and weighed. Calcium sulphate is found by determining the lime in the acetate solution. Corrections are made for the solubility of calcium fluoride in acetic acid (0·002 g. per 1 g. of sample) and in the ammoniacal acetate solution (0·0025 g. per 1 g. of sample).

A. R. Powell.

Preparation of cyanides from calcium cyanamide and dicyanodiamide. A. E. Kretov (J. Chem. Ind. [Russia], 1925, **2**, 350—352, 482—484; Chem. Abstr., 1926, **20**, 3334).—In the preparation of sodium cyanide by fusing calcium cyanamide with sodium carbonate or chloride, an excess of the carbonate or chloride is desirable to lower the m.p., since the reactions are reversible. From calcium cyanamide (50% purity, 15·5% N) 80% of the nitrogen is recoverable as pure cyanide when equal weights of carbonate and calcium cyanamide are heated in an electric furnace for 25—30 min., and the aqueous extract is filtered. Hydrogen cyanide is then distilled into an alcoholic solution of an alkali. Only 50% of the nitrogen of dicyanodiamide can be utilised. The dicyanodiamide is obtained by boiling 1 kg. of calcium cyanamide with 2 litres of water for 30—40 min., filtering, and crystallising, the mother-liquor being used again. Lime is removed from the boiling liquid by carbon dioxide; if aluminium sulphate is added to the boiling liquid the yield may reach 95% of the theoretical. By fusing dicyanodiamide with sodium carbonate and carbon (168 : 212 : 9) at 500°, only cyanates are obtained; cyanides are best obtained by heating at 750° for 20—30 min.

A. A. Eldridge.

Solubility of potassium bisulphite. J. H. Platt and (Miss) D. Hudson (J. Soc. Dyers and Col., 1926, **42**, 348—349).—The following values are given for the solubility of potassium hydrogen sulphite (recrystallised commercial potassium metabisulphite) in water, expressed in g./100 g. of water:—15°, 45·5; 25°, 51·5; 50°, 67·4; 60°, 76·5; 70°, 85·6; 75°, 91·5.

R. Brightman.

Manufacture of sulphur from sulphurous gas obtained as a by-product in refining metals. N. F. Yushkevich and V. A. Karzhavin (J. Chem. Ind. [Russia], 1925, **2**, 474—478, 559—563; Chem, Abstr., 1926, **20**, 3335).—On reduction of sulphur dioxide (from copper smelting) by charcoal and coke at 800°, 99% should form sulphur; the yield should decrease with rise of temperature, being 82·6% at 1400°. The gaseous mixture obtained should contain 19·8 or 16·0% of sulphur vapour at 800° or 1400°, respectively. The anticipated results were confirmed between 800° and 1180° by passing mixtures of carbon monoxide and sulphur dioxide through a silica tube containing a catalyst composed of pumice saturated with ferrous ammonium sulphate solution and calcined in dry air at 1000°. The catalyst was first heated in carbon monoxide at 900°, and then in a mixture of sulphur dioxide and carbon monoxide to produce ferrous sulphide. A more active catalyst is, however, desirable.

A. A. Eldridge.

Patents.

Catalytic process for producing concentrated and fuming sulphuric acid. S. G. S. Dicker. From K. Kudoh (E.P. 258,974, 17.7.25).—The process is carried out in a closed circuit of apparatus preferably using pyrites cinders as a catalyst. The mixture of sulphur dioxide and oxygen, raised to reaction temperature by heat from the catalysis chamber, is catalysed and the sulphur trioxide produced is removed by absorption in sulphuric acid and replaced by an addition of sulphur dioxide mixture corresponding to that catalysed to sulphur trioxide. The gaseous mixture, which is heated by gases leaving the apparatus, is recirculated, the composition of the reacting mixture varying according to the activity of the catalyst.

H. Royal-Dawson.

Production of sulphuric acid. M. S. Stutchbury. From Metallbank und Metallurgische Ges. A.-G. (E.P. 260,047, 22.7.25; cf. B., 1921, 693; 1922, 982).—Mechanical mixing and washing chambers are alternately employed in the front of the plant, the rear consisting only of washing chambers. Arranged outside the double-cycle plant is a third washing chamber supplied with nitrosylsulphuric acid of about d 1·71, which is denitrated by fresh hot gases in the foremost chamber, and serves for absorbing the oxides of nitrogen. The plant is modified by placing a mechanical denitrating device in the front of the first washing chamber to denitrate the daily production of acid when using cold sulphur dioxide gases, while at the same time it supplies a partially or wholly denitrated acid to the last washing chamber.

H. Royal-Dawson.

Manufacture of acetic acid. H. Dreyfus (E.P. 259,641, 13.6.25).—By passing methyl formate vapour, under pressure and at relatively high temperature, in contact with a catalyst at 400—450°, such vapour is continuously isomerised to acetic acid, which distils off and is separated from the resulting vapour mixture by fractional condensation. Such catalysts are the oxides of copper, tin, lead, or zinc; acetates of copper or zinc; methoxides of tin, zinc, aluminium, etc.; or mixtures of these with potassium or sodium acetate. The reaction takes place, with advantage, at 200—300° under a pressure of 50—150 atm. F. G. Crosse.

Manufacture of metallic nitrides in admixture with lithium nitride and amide. Soc. d'Etudes Minières and Industrielles (E.P. 245,762, 31.12.25. Conv., 8.1.25).—Nitrides of iron, nickel, cobalt, or molybdenum are obtained by heating the metal in question in nitrogen while admixed with lithium nitride or amide, the reaction taking place at a pressure of $<$250 kg./sq. cm. and a temperature of 500—600°. H. Royal-Dawson.

Preparation of cream of magnesia. G. C. Hurrell (E.P. 258,134, 10.12.25).—An aqueous suspension of magnesium oxide is heated to 50—80° for a time varying with the physical condition of the magnesia, light amorphous oxide requiring a few minutes only, while denser and more crystalline types of "light magnesia" need several hours' heating. The mixture is treated while hot in a colloid mill producing an intense dispersing effect. About 95% of the resulting gel is magnesium hydroxide, and this can be increased by keeping the temperature raised for some hours. W. G. Carey.

Manufacture of lime. A. Stephenson, and Allen-Liversidge, Ltd. (E.P. 258,661, 24.6.25).—The excess of water is removed from the calcareous residue obtained in the manufacture of acetylene, and the extracted material calcined at sufficiently high temperature to remove the uncombined water and produce calcium hydroxide in a dry state. H. Royal-Dawson.

Manufacture of solid mixtures of alkali hypochlorite and alkali chlorides. A. Oppé (E.P. 258,821, 17.6.26. Conv., 17.4.26).—Chlorine is made to act upon dry alkali hydroxide suspended in a neutral liquid, *e.g.*, carbon tetrachloride. H. Royal-Dawson.

Process of producing hydroxides and carbonates. M. Buchner (E.P. [A] 235,588 and [B] 255,474, 12.6.25. Conv., 14.6.24).—(A) Fluorides and carbonates or hydroxides, the bases of which give insoluble fluorides, are treated at 80° under pressure in the presence of a small quantity of water insufficient to produce a solution, or sufficient only to produce a saturated solution. The reaction may be carried out in the presence of carbon dioxide or of substances evolving the gas. (B) Fluorides the bases of which yield insoluble carbonates and hydroxides are treated under pressure with carbonates and hydroxides, the bases of which yield insoluble fluorides. H. Royal-Dawson.

Production of barium oxide and/or hydroxide. B. P. Hill, and Blaydon Manure and Alkali Co. (1877), Ltd. (E.P. 259,395, 30.10.25).—These products are obtained by introducing barium carbonate, in the form of a suspension or cloud, into a chamber where it becomes heated by direct contact with the gases of combustion from a flame or from oil burners. Alternatively, the barium carbonate may be mixed with finely divided coal and blown into the furnace. The solid products are collected and extracted with hot water. F. G. Crosse.

Producing chemically pure sodium chloride from natural brine. C. S. Robison, Assr. to Mulkey Salt Co. (U.S.P. 1,598,935, 7.9.26. Appl., 29.1.25).—Brine liquor containing foreign precipitable matter is evaporated to precipitate the heavier sodium chloride crystals, the lighter foreign matter in suspension being removed before the supersaturation point. H. Royal-Dawson.

Manufacture of chromates. W. Carpmael. From I.-G. Farbenind. A.-G. (E.P. 259,447, 8.2.26).—Chrome ore in grades of which the coarse is such that 15—30% of the total ore would remain on a screen of 4900 meshes/sq. cm. is mixed with sodium carbonate and heated in a revolving furnace to 1100°. After quenching, the chromate liquor is leached out. The residue is treated with sulphuric acid (d 1·116) at 50°, so that not all the iron and magnesium oxide are dissolved, and the slightly basic liquor run off, while the residue is dried and again used in the first part of the process. F. G. Crosse.

Manufacture of iron carbonyl. J. Y. Johnson. From Badische Anilin & Soda Fabrik (E.P. 259,407, 13.11.25).—Iron from which the film of oxide has been completely removed is treated with carbon monoxide free from oxidising gases at temperatures lower than those at which carbon monoxide would be decomposed to carbon dioxide and carbon. Thus pure commercial iron powder, obtained by the reduction of iron oxide, is treated for several hours with dry hydrogen at 200° under a pressure of 130 atm. and, subsequently, with a current of carbon monoxide at 150 atm. pressure and 200°; 90% of the iron is transformed into carbonyl in 6 hrs. Without the hydrogen treatment only 20% is converted. F. G. Crosse.

Producing metal compounds. A. F. Meyerhofer (E.P. 245,719, 253,149, and 253,150, 8.5.25. Conv., 10.1.25).—(A) A silicofluoride or borofluoride is decomposed by heat into a metal fluoride and flouride of silicon or boron, and the metal fluoride is treated with a salt (other than carbonate) of the acid to be introduced, whose base yields a sparingly soluble fluoride. The metal fluoride produced is used with silicon or boron fluoride and an appropriate salt, with or without the aid of acid, to re-form silicofluoride or borofluoride (cf. E.P. 222,838; B, 1925, 670). Thus sodium silicofluoride is heated to yield sodium fluoride, which is converted by means of calcium nitrate into calcium fluoride and sodium nitrate, the former being converted by means of silicon fluoride and sodium chloride into silicofluoride. Sulphides, sulphites, or phosphates may be similarly produced. (B) Complex fluorides other than silicofluorides or borofluorides are used, as, *e.g.*, titanofluorides. (C) Barium silicofluoride is treated as before, but the sparingly soluble fluoride and the silicon fluoride are used in the presence of acid to form hydrofluosilicic acid, the barium silicofluoride being

reproduced (cf. E.P. 226,491 ; B, 1925, 714). An important application of this is in the production of hydrogen peroxide. F. G. CROSSE.

Compounds of copper for use in combating fungi. C. A. NEWHALL (U.S.P. 1,598,982, 7.9.26. Appl., 31.10.23).—Sodium hydroxide is added to a solution of copper sulphate, and the basic salt precipitated is heated in the mother liquor to 60°, filtered, and rapidly dried. A bulky basic salt of copper is obtained. Seed grain may thereby be given adequate protection against fungi. T. S. WHEELER.

Neutralising the free acid in commercial ammonium sulphate. COKE & GAS OVENS, LTD., and R. PEARSON (E.P. 259,669, 14.7.25).—The sulphate is conveyed, during treatment, through a zig-zag path, by means of three parallel worm conveyors placed horizontally one above the other in respective compartments of a common chamber. Ammonia gas is passed through the three compartments in the reverse direction, steam passing simultaneously through the outer chamber. The product obtained is thus dried, neutralised, and powdered. Soda ash or other alkaline material may replace ammonia gas by a simple adaptation of the process. F. G. CROSSE.

Making sulphite cooking liquors. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,599,488, 14.9.26. Appl., 19.7.22).—The sulphur content of digester relief liquor is recovered by precipitation as an insoluble monosulphite, whilst avoiding the co-precipitation of any substantial quantity of organic matter. The monosulphite is subsequently converted into bisulphite by sulphurous acid, and used as a cooking liquor.
D. J. NORMAN.

Separating carbon monoxide from industrial gases. A. A. L. J. DAMIENS (E.P. 259,704, 6.8.25. Addn. to 230,106, B., 1925, 847).—The carbon monoxide is absorbed under a slight pressure, and a cupric derivative is used in the presence of a reducing agent capable of bringing it to the cuprous state, *e.g.*, cupric sulphate with metallic copper. F. G. CROSSE.

Recovery of sulphur dioxide from blow-pit gases. G. A. RICHTER and W. B. VAN ARSDEL, Assrs. to BROWN Co. (U.S.P. 1,599,490, 14.9.26. Appl., 22.4.25).—The gases and steam are passed countercurrent to, and in direct contact with, relatively cold water, so that the gases are partly cooled and only a portion of the steam is condensed. Further cooling of the gases and the removal of another portion of the steam is effected by passing them through towers packed with relatively cool, inert, interstitial material.
D. J. NORMAN.

Concentration of diluted nitrous gases. H. JOHNSEN, Assr. to NORSK HYDRO-ELEKTRISK KVAELSTOFAKT. (U.S.P. 1,600,547, 21.9.26. Appl., 6.5.25. Conv., 15.5.24).—The gases are absorbed in a solution of an alkali metal phosphate, and the mixture is heated in a closed chamber to such a degree that the nitrogen escapes in the form of nitrogen oxides with the reformation of the alkali phosphate at the same time.
H. ROYAL-DAWSON.

Production of hydrocyanic acid. G. BREDIG and E. ELÖD, Assrs. to R. KOEPP & Co. (U.S.P. 1,598,707, 7.9.26. Appl., 21.11.24).—See E.P. 229,774 ; B., 1925, 315.

Deoxygenating air by the combustion of sulphur or its incompletely oxidised compounds. T. CHMURA (E.P. 237,257, 14.7.25).—See F.P. 603,989 ; B., 1926, 742.

Recovery of cyanide [from ammoniacal gas liquor] (U.S.P. 1,600,228).—See II.

VIII.—GLASS ; CERAMICS.

Mechanism of spalling [of pottery]. F. H. NORTON (J. Amer. Ceram. Soc., 1926, **9**, 446—461 ; cf. B., 1925, 171).—The stresses developed in a solid body when subjected to rapid heating and cooling were studied by the photoelastic method, bakelite specimens being used. The stresses set up by sudden heating were of the same order as, but opposite in sign to, those caused by cooling through the same range of temperature. Spheres and bricks of fine grog and fireclay were fired to a vitreous state and then subjected to similar heat treatment. An examination of the cracks produced in these specimens indicated that spalling fractures were caused both by shear and by tension stresses ; spalling caused by rapid heating was due entirely to shear stresses, whereas cooling fractures were due mainly to tension stresses and, to a less degree, to shear stresses ; sudden heating produced no large tension stresses ; both heating and cooling produced approximately equal shear stresses, corresponding in value to about one-half the maximum tension or compression strain ; shear fractures occurred near the edges and corners of the specimen, and cracks entered the surface along the lines of shear, *i.e.*, at about 45° to the surface ; tension fractures appeared across the bricks remote from the corners, due to concentration of tension strains at these points. Torsion tests were made on a number of firebricks at temperatures up to 1550°. The bricks gave evidence of plastic flow at approximately the temperature at which they showed initial collapse under the standard load test. F. SALT.

Rupture of glass. F. W. PRESTON (J. Soc. Glass Tech., 1926, **10**, 234—269).

PATENTS.

Annealing furnaces. THE UNITED GLASS BOTTLE MANUFACTURERS, LTD., Assees. of E. A. C. PRYOR (E.P. 259,629, 21.4.25). — An annealing furnace is constructed with an upper chamber which is heated to the temperature of articles introduced for annealing on a heated endless conveyor, and a lower chamber in which the portion of the conveyor not in use is heated. By these means, and by constructing the walls of insulating material, the heat of articles is conserved so that they are annealed substantially by their own initial heat.
W. G. CAREY.

Manufacture of ceramic bodies. F. H. RIDDLE (E.P. 259,757, 9.10.25).—In the production of ceramic ware having a high dielectric strength and high heat resistance a lower temperature of firing is sufficient if mineral anhydrous aluminium silicates of the sillimanite

group, other than natural sillimanite itself, be used. The best results are obtained with andalusite, or andalusite and artificial sillimanite, or andalusite and zirconium silicate. A small amount of barium sulphate or talc increases the insulating value, and the addition of fused magnesium oxide or titanium compounds improves the texture of the ware. W. G. CAREY.

Abrading materials in the form of slabs, wheels, discs, etc. P. M. SALERNI (E.P. 259,679, 16.7.25).—Powdered carborundum or emery is mixed with an adhesive such as rubber dissolved in a solvent, with sulphur added for vulcanising. The mixture is heated sufficiently to evaporate the solvent, and is then treated in a vulcanising apparatus at 165° for 1 hr. The friable mass is broken up, compressed and heated simultaneously in a mould, and subjected to a final compression in a mould of true shape. Since no baking or vulcanisation takes place after compression, distortion is avoided. Adhesives such as shellac or other lac or resin may be used instead of rubber. W. G. CAREY.

Abrasive cement. H. O. KEAY (E.P. 260,171, 29.4.26).—A mixture of sand, a synthetic resin, furfuraldehyde, and alcohol. B. W. CLARKE.

Forming sheet glass. L. MELLERSH-JACKSON. From HARTFORD-EMPIRE Co. (E.P. 259,150, 31.5.26).

Manufacture of sheet glass. E. C. R. MARKS. From ERIE GLASS Co. (E.P. 258,093, 28.9.25).

Apparatus for the manufacture of sheet glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (E.P. 253,866, 8.4.26. Conv., 20.6.25).

Manufacture of plate glass etc. C. HEUZE (E.P. 249,863, 23.3.26. Conv., 28.3.25).

Lehrs for annealing glassware. W. J. MELLERSH-JACKSON. From HARTFORD-EMPIRE Co. (E.P. 260,041, 22.7.25).

Electric glass-annealing lehrs. V. F. FEENY. From ILLINOIS PACIFIC GLASS Co. (E.P. 260,104, 30.10.25).

IX.—BUILDING MATERIALS.

Physical transformations undergone by cement during setting. C. ZAMBONI (Giorn. Chim. Ind. Appl., 1926, **8**, 469—472).—The changes in sp. gr. and in the quantity of water uniting with cement during setting, show that the quality of a cement depends on its ability to combine with a large proportion of water in minimum time, this determining its compactness, and hence its strength. The rapidity of hardening increases with the fineness of the cement, and the quality of the latter depends largely on the system of burning employed. Since the difference between the sp. gr. of an aggregate and that obtaining after this has hardened in water for a given time determines its strength, it is possible to obtain a rapid measure of the intrinsic value by two measurements of the sp. gr. at a short interval. It is not possible to fix for cement standards for all the necessary chemical characteristics. T. H. POPE.

Determination of soluble silica in cements, mortars, and concretes, and of the cement content of mortars and concretes. D. FLORENTIN (Ann. Chim. analyt., 1926, [ii], **8**, 321—324).—See B., 1926, 668.

PATENTS.

Cementitious compositions. G. J. FINK, Assee. of J. A. MCCORMICK and C. A. CABELL (E.P. 239,504, 24.8.25. Conv., 5.9.24).—Hydrated lime is mixed with about 4% of a material of the "ciment fondu" type, *e.g.*, calcium aluminate; 0·5% of a sulphate, *e.g.*, aluminium sulphate; 5% of a slightly soluble carbonate, *e.g.*, calcium or magnesium carbonate; and about 0·3% of sugar as a retarder. A cementitious material with quick initial setting properties, relatively high early and final strengths, and a high degree of workability is produced. B. W. CLARKE.

Waterproofing Portland cement concrete. E. C. E. LORD (U.S.P. 1,599,903, 14.9.26. Appl., 14.12.25).—Paraffin emulsified with kerosene and soap-water is introduced into the gauging water in order to prevent the ingress of water or other liquids after the concrete has hardened. B. W. CLARKE.

Restoring plaster of Paris to its original state after use. A. E. LOVATT, F. M. MITCHELL, and E. A. MITCHELL (E.P. 260,110, 11.10.25).—Used plaster of Paris, *e.g.*, in the form of moulds, is first cleaned by saturating the exterior surface with water, drying, and removing the surface coating, and then reduced to granules of uniform size, which are subjected to a critical heat treatment in an externally heated chamber (usually to 170° for about 30 minutes). B. W. CLARKE.

Production of highly porous clay material. E. I. LINDMAN (E.P. 256,580, 21.6.26. Conv., 6.8.25).—A quaternary clay is first quickly heated to a temperature near its m.p. (1000—1500°) in a shaft furnace, and the product then cautiously heated, in a rotary furnace, to such a temperature that the material assumes a "thoroughly viscous melting consistency," to facilitate the expansion of the material by the enclosed gases, whereupon the heating is stopped. The product thus obtained has a sp. gr. generally below 0·5, is highly insulating, cheap, and can be usefully employed as an admixture in concrete. F. G. CROSSE.

Composition for the manufacture of bricks, tiles, artificial stone, etc. T. N. BACKHOUSE and H. OLIVER (E.P. 260,074, 22.8.25).—The composition described is composed of 5% by volume of calcined magnesite (or magnesium oxide), 2·5% by volume of powdered magnesium chloride, and 92·5% by volume of filling material such as sand, calcined clinker, ashes, slate, etc. F. G. CROSSE.

Hardening and preserving natural and artificial stones. D. DE ROS and F. BARTON (E.P. 260,031, 18.7.25).—A volatile silicon compound is sprayed on to the wetted surface of the stone, forming insoluble silicon compounds or a thin film of silica which hardens and preserves the stone without affecting its appearance. Silicon tetrachloride is suitable for non-calcareous stones, but the tetrafluoride is to be preferred with dolomitic stones. B. W. CLARKE.

Treatment of stone, metal, or other materials in order to clean and/or preserve them. J. E.

MARSH (E.P. 260,331, 23.7.25).—An alkaline solution and hydrogen peroxide, or sodium peroxide, is used.
F. G. CROSSE.

Wall coverings. G. E. HEYL (E.P. 259,826, 17.3.26).—Fibrous material, *e.g.*, paper or air-dry wood pulp, is disintegrated first in a dry state in a high-speed impact beater mill and then with the addition of oil or water or both, and 5—15% of a lubricant filler such as china clay, barytes, or gypsum, in a high-speed colloid mill. Glue, pigments, cement, etc. can be added to the liquid product, which is used in the wet condition as a wall-covering material. B. W. CLARKE.

Production of fire-proof bricks. S. E. SIEURIN (E.P. 259,805, 29.12.25).—Silicon carbide is intimately mixed with a binding agent of finely divided silicic acid and fire-clay or lime. The amount of silicic acid in the binding agent must be at least 80% by weight if clay is used, or 90% when lime is used. The proportions of silicon carbide and binding agent may vary from about 30 to 90% of the former, a 50% content giving a brick with a softening temperature of about 1670°.
W. G. CAREY.

Production of ash or clinker from town and like refuse. G. E. HEYL (E.P. 260,182 31.5.26).—Refuse is calcined in a rotary retort at a temperature not exceeding the fusion temperature of the ash or clinker produced, which is subsequently ground.
B. W. CLARKE.

Production of floor coverings. K. SCHNEBLE (E.P. 249,569, 23.3.26. Conv., 23.3.25).

Fibre and cement composition for use as a mortar or plastic composition for walls etc. J. MELANDRI, and SPUN CONCRETE CONSTRUCTION CO. LTD. (E.P. 260,427, 5.11.25).

Packing powdered material [cement] (E.P. 259,299, 7.7.25).—See I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Blackening and burnishing iron. MEYSAHN (Chem.-Ztg., 1926, **50**, 693).—To blacken iron, it is freed from grease, coated with a thin layer of linseed or other oil, linseed oil varnish or ozokerite, and slowly heated, the process being repeated if necessary. The surface obtained is both lasting and resistant to the action of air, water, acids, and alkalis. In burnishing, the iron is cleaned with acid, treated with various substances according to the colour required, and subsequently dried in the air and polished. A blue shade is obtained with ferric chloride and potassium ferrocyanide or sodium hyposulphite and lead acetate. The vapour from a heated mixture of hydrochloric and nitric acids gives a brown shade, and a solution of tannic and acetic acids a black one. F. R. ENNOS.

Abnormal absorption of gases by steel. T. C. SUTTON and H. R. AMBLER (Trans. Faraday Soc., 1926, **22**, 406—419).—When a quantity of cordite is mixed with steel filings and exploded in a closed container the volume of gas produced is much less than the theoretical. The difference is due to adsorption of gas by the fine fragments of steel at the high temperature and pressure produced by the explosion. On subsequently heating the steel *in vacuo* no evolution of gas occurs until a temperature of 600° is reached; above this the rate of evolution increases with rise of temperature, and is particularly rapid at 1000°. The amount of gas adsorbed by the steel may be as high as 30 c.c./g. In the process of desorption carbon dioxide is first evolved, followed by carbon monoxide and hydrogen as the temperature rises; the nitrogen adsorbed cannot, however, be removed by heating to 1000° and appears therefore to be retained as nitride. The energy evolved by the adsorption of 1 c.c. of the mixed gases is of the order of 0·75 g.-cal.
A. R. POWELL.

Magnetic permeability of cold-drawn steels. G. DELBART (Compt. rend., 1926, **183**, 662; cf. B., 1926, 194).—The magnetic permeability of a steel containing 0·300% C, 0·084% Si, 0·024% S, 0·027% P, and 0·850% Mn has been studied after annealing at 15°, 700°, 850°, and 980°. At 700° a globular pearlite structure was observed, giving place almost entirely to a sorbitic pearlite structure at 850° and 980°. This change is accompanied by an increase in the magnetic permeability, particularly in fields of from 7 to 20 gauss, where the maximum values are obtained. For magnetic fields up to 150 gauss the permeability is higher for 700° than for 15°, but decreases at higher temperatures.
J. GRANT.

Properties of mild steel at high temperatures. E. J. RANG (Inst. Civil Eng., Selected Papers, 1926, No. 42, 12 pp.).—With increasing carbon content the limit of proportionality of steels becomes indefinite. It is concluded that during the extension of high-carbon steel test bars, the elastic limit of the steel is being reached continually, a small amount of work done on the metal, and a new elastic limit formed. At temperatures above 860° no common steel possesses any elasticity, and a beam will be deflected considerably by its own weight if the temperature is maintained. The expansion of steel when heated is usually shown as a linear curve, but actually a decrease in length occurs when approaching 760°. The actual expansion is dependent to some extent on the carbon content at temperatures above 760°, and is apparently independent of that factor at lower temperatures. C. A. KING.

Caustic embrittlement of steel. S. W. PARR and F. G. STRAUB (Chem. Met. Eng., 1926, **33**, 604—607).—Cracks occurring in boilers using alkaline water are characteristically different from ordinary corrosion or fatigue cracks. They do not follow the line of probable maximum stress, but run irregularly along grain boundaries from one rivet hole to another, never extending into the body of the plate beyond the lap of the seam. Sodium carbonate predominated over sodium sulphate in all waters examined from boilers suffering from embrittlement, and in use would develop a caustic reaction due to sodium hydroxide. Corrosion tests, in which strips of steel were maintained under strain in the test solution, showed that sodium hydroxide solution must have a concentration of about 35%, and, at the same time, the stress in the metal must be above the yield point to produce rupture in less

than a month. Neither sodium carbonate nor sodium sulphate caused rupture, even at 50% concentration and higher stress, but when present together with an injurious amount of sodium hydroxide they tended to prevent embrittlement. When the ratio Na_2CO_3 (Na_2SO_4) : NaOH was >2 : 1, rupture did not occur with a 40% sodium hydroxide solution. In practice high concentrations of sodium hydroxide are developed between the laps of the plate and under the rivet heads, a value of 350,000 pts./million being thought possible. Embrittlement may be prevented by converting part of the sodium carbonate into sulphate, by using either sulphuric acid or aluminium or magnesium sulphate.

C. A. King.

Cementation of iron by silicon chloride. A. Sanfourche (Compt. rend., 1926, **183**, 791—793).—Cementation without solid contact was brought about by the action of silicon chloride on compact iron in the form of cylindrical bars of soft iron (0·2% C). In the first set of experiments the vapour of silicon chloride was driven, by means of a current of nitrogen, over the bars heated in a silica tube. Reaction set in at 800°, a superficial layer of a ferrosilicon alloy being formed, the thickness of which increased with rise of temperature and increase in the duration of the experiment. In the second set of experiments the vapour was driven over the bars by means of a current of hydrogen; in this case the action was more intense but less regular. Microscopical examination showed a sharp demarcation between the layer of alloy and the unaltered metal. When nitrogen was used the two layers were separated by a thin layer very rich in graphite formed by concentration of carbon displaced by silicon from the cementite eutectoid. When hydrogen was used this graphitic layer was not always present. The layer of alloy never contained more than 14% Si. The compound Fe_3Si requires 14·34% Si, and reasons are given for supposing this compound to be present.

M. E. Nottage.

Resistance of metals [and alloys] to nitric acid. J. G. Thompson (Chem. Met. Eng., 1926, **33**, 614—616).—For equipment for nitric acid production, aluminium and its alloys proved satisfactory in its resistance to 100% nitric acid, being superior to all the iron alloys tried, with the exception of a high-silicon cast iron. They were, however, quite unsuitable for concentrations less than 68%, being severely corroded and deeply pitted. Molybdenum steel or steel containing 5·5% Cr was little better than plain steels, but increase in the chromium content to 10·8—15·7% effected marked improvement in its resistance to the stronger acid, whilst steels containing over 16% Cr showed no signs of attack by nitric acid up to 68% concentration. The presence of nickel tends to reduce the beneficial effect of the chromium. Metallic chromium might be used as a plating material on more easily attacked alloys up to 68% concentration of acid. Of all the alloys tested, a high-silicon cast iron (Si 14·55%, C 0·59%) proved the most resistant. It was apparently unattacked by acid of any concentration; but a somewhat similar cast iron (Si 12·24%, C 1·17%) was less satisfactory.

C. A. King.

Analysis of dental gold alloys. W. H. Swanger (U.S. Bureau of Standards, Sci. Paper 532, 1926, **21**, 209—239).—A scheme for the analysis of dental alloys is described. The alloy is dissolved in aqua regia, silver chloride and iridium are filtered off and determined separately. By addition of sodium acetate to the filtrate, followed by boiling, stannic and ferric hydroxides are completely separated from the residual metals, and are then determined by usual methods. After precipitating gold from the solution by reduction with sulphurous acid (traces of platinum and palladium are removed from this precipitate by redissolving and reprecipitating gold with oxalic acid), palladium is determined as the dimethylglyoxime compound. The solution is evaporated to dryness, excess of dimethylglyoxime is destroyed with nitric acid, and sulphurous acid is added. Copper is then precipitated as cuprous thiocyanate. Platinum and rhodium are removed as sulphides from the residual solution, and are then determined by the method described by Wichers (A., 1924, ii, 706; B., 1926, 852). Zinc, nickel, manganese, and magnesium in the filtrate from platinum and rhodium are determined by the usual methods.

L. M. Clark.

Rate of tarnishing of copper–gold alloys in oxygen, carbon dioxide containing hydrogen sulphide, and in air containing iodine. G. Tammann and W. Rienäcker (Z. anorg. Chem., 1926, **156**, 261—274).—A detailed discussion of the mechanism of the tarnishing of alloys of copper and gold of varying composition in atmospheres of the gases in question. The effect of raising the temperature is also examined.

M. Carlton.

Manufacture of sulphur from sulphurous gas obtained as a by-product in refining metals. Yushkevich and Karzhavin.—Sec VII.

Coatings on aluminium or light alloys. Cournot and Bary.—See XI.

Separation of gold from silver. Vanioukov.—See XI.

Patents.

Malleable cast iron. J. E. Fletcher, J. G. Pearce, and The British Cast Iron Research Assoc. (E.P. 260,070, 15.8.25).—The physical properties of malleable iron castings are improved by making the grain-size of the original castings as fine as possible, either by the use of moulds of relatively high thermal conductivity or by the addition to the metal of an element such as manganese, sulphur, chromium, molybdenum, or tungsten, which promotes the stability of carbides, and opposes graphitisation. Both methods may be employed together. The grain-size initially present in the cast metal persists throughout the annealing process.

M. Cook.

Melting iron and other metals. Y. A. Dyer (E.P. 260,143, 25.1.26).—Air or oxygen, preheated if necessary, is forced, at a pressure of ¼—1½ lb./sq. in., through a detached combustion chamber, and the hot gases are conducted to the lower portion of a shaft furnace or cupola. Alternate thin layers of carbonaceous fuel and relatively thick layers of metal are charged into the cupola. Secondary air or oxygen may be admitted to

the gases produced by combustion at any point between the combustion chamber and the reduction furnace.
C. A. KING.

Steel. J. C. McGUIRE (U.S.P. 1,599,425, 14.9.26. Appl., 17.8.25).—The preparation of a steel containing C 1·4%, W 4%, Cr 11·5%, Ti 0·3%, Ni 0·85%, Si 0·35%, Mn 0·23%, P 0·025%, S 0·025%, and Fe 81·32% is described. C. A. KING.

Hardening of steel. P. M. SALERNI (E.P. 260,026, 16.7.25).—The heat treatment of steel bodies for hardening purposes is controlled automatically by the loss or change in the magnetic properties of the steel at its point of decalescence. This is effected by using an electro-magnet to hold the steel in position in the furnace until the change in magnetic properties releases the steel, which then falls out of the furnace into a suitable quenching bath. A supporting device may partly counterbalance the weight of the steel in the furnace.
C. A. KING.

[" Rustless "] iron alloy. H. G. FLODIN and E. G. T. GUSTAFSSON (E.P. 243,761, 28.11.25. Conv., 29.11.24).—An alloy of iron possessing a high degree of resistance to rusting contains 0·25—8·99% Cr, and up to 0·05% C.
C. A. KING.

Coating cast-iron with lead. O. SPENGLER (E.P. 260,452, 15.1.26).—The cast-iron to be coated is first pickled, and then treated with a solution of a metallic salt from which the metal is precipitated by iron, *e.g.*, copper sulphate. The layer of copper so formed is coated with lead by painting it with a solution of a zinc halide and a tin salt, which is heated, and a piece of lead dropped on it. When the surface is sufficiently hot the lead flows smoothly, giving a homogeneous coating. F. G. CROSSE.

Manufacture of aluminium alloy. P. BERTHÉLEMY and H. DE MONTBY (U.S.P. 1,599,869, 14.9.26. Appl., 16.6.25).—A mixture of copper, manganese, ferrosilicon, tungsten, magnesium, and aluminium is melted in a magnesia-lined plumbago crucible containing a mixture of wood charcoal, calcium fluoride, magnesium oxide, and arsenious acid. When scoured and cast into ingot form this rich alloy is subsequently mixed with pure aluminium. M. COOK.

Improving metal castings. M. ESTERER (E.P. 259,624, 7.4.25).—During solidification the arrangement of the molecules of a metal casting is influenced by vibrations produced mechanically, or by electrical or magnetic oscillating fields of high frequency. Both methods may be used concurrently. Experiments have shown that liquid metals in the process of solidification crystallise differently under the influence of vibrations of certain definite wave-length, and the appropriate frequency for any particular metal or alloy can be determined only by experiment. C. A. KING.

Refining nickel and nickel-copper mattes. O. LELLEP, Assr. to INTERNATIONAL NICKEL CO. (U.S.P. 1,599,424, 14.9.26. Appl., 7.4.23).—In the process of eliminating sulphur from nickel or nickel-copper mattes, the molten matte is treated with a blast of air, and the necessary additional heat for the reaction is supplied.
C. A. KING.

Cleaning metal [iron and steel] by electrolysis. I. H. LEE, Assr. to STUDEBAKER CORP. (U.S.P. 1,598,731, 7.9.26. Appl., 1.5.25).—The article to be cleansed forms the cathode of an electrolytic cell, in which the electrolyte is a dilute (3%) solution of sodium oxalate, citrate, or tartrate, with about 0·5% of sodium hydroxide. The anode is also of iron, and a current of 75 amp./sq. ft. of surface to be treated is led through the cell at 75°.
T. S. WHEELER.

Removal of deposit, scale, or incrustations from metal. G. RÜTSCHE (E.P. 253,073, 15.3.26).—Incrusted metal is subjected to the blast of a large flame produced by burning a preheated mixture of oxygen and a low-grade mineral oil in an excess of oxygen. The tension difference set up between the metal and scale causes the latter to split and crack, and the loosened crust to be blown away by the flame. The best results are obtained when the metal is heated to 100° prior to the application of the flame. M. COOK.

Ferrous alloy. B. D. SAKLATWALLA (U.S.P. 1,599,435, 14.9.26. Appl., 26.8.22).—See E.P. 202,971 ; B., 1923, 600 A.

Treatment of lead-zinc sulphur ores, mattes, etc. E. A. ASHCROFT (U.S.P. 1,599,269, 7.9.26. Appl., 28.6.23. Conv., 2.6.23).—See E.P. 210,011 ; B., 1924, 262.

Concentration of ores. A. B. EMERY (U.S.P. 1,599,561, 14.9.26. Appl., 5.12.22. Conv., 21.9.22).—See E.P. 204,302 ; B., 1924, 1370.

Continuous tin plate furnaces. J. J. JONES (E.P. 241,589, 19.10.25. Conv., 18.10.24).

[Composition for] affixing metallic powders to articles or surfaces. W. BRADLEY and W. H. J. RATTEW (E.P. 259,483, 10.4.26).

XI.—ELECTROTECHNICS.

Electrolytic coatings on aluminium and light alloys, their adherence and resistance to corrosion by sea-water. J. COURNOT and J. BARY (Compt. rend., 1926, **183**, 789—791).—Aluminium and duralumin were coated with electrolytic deposits of cadmium, cobalt, and chromium, after an initial deposit of copper ; cadmium was also deposited directly on duralumin. The initial cleaning of the metal is most important ; the method finally adopted consisted in sand-papering the metal, washing it with water, passing it rapidly through a bath of nitric acid of *d* 1·14, and again washing. In certain cases, the nitric acid bath was replaced by one containing sodium bicarbonate and chloride. The composition of the electrolyte and conditions for deposition are given for coatings of copper, cadmium, cobalt, and chromium. The adherences of all deposits were satisfactory so long as the precautions of manipulation and cleaning were rigorously observed. The sheet metal used was 1·6 mm. thick. Copper deposited alone on aluminium adhered well, and showed little tendency to scale ; the adherence was not quite so good on duralumin, and the tendency to scale was greater. Cobalt, deposited after a normal initial coating of copper, and chromium, after a light initial coating of copper, adhered well on both aluminium and duralumin. Cadmium adhered

well to aluminium after an initial light coating of copper, but in the case of duralumin the best results were obtained by depositing cadmium directly on to the metal. The resistance of all these deposits to corrosion by artificial sea-water was found to be most pronounced at the level of the liquid, when the test-piece was not completely submerged; the resistance to corrosion was good for the part completely submerged. In certain cases surface-hardening, due to the effect of electrolysis as well as to the hardness of the deposited metal, was observed. M. E. NOTTAGE.

Batch process for the electrolytic separation of gold from silver. V. A. VANIOUKOV (Messager ind. Métaux [Russia], 1920, [7—8]; Chem. Abstr., 1926, **20**, 3269).—In an improved Balbach apparatus, bars of auriferous silver are placed in wooden tanks in the electrolytic bath in which silver T-shaped anodes are placed over, and in contact with, the auriferous silver, thin silver sheets in the bottom of the bath serving as cathodes. When a bath containing 2—4% of silver nitrate and 1·5—2% of free nitric acid was employed, the loss of silver was only 0·026—0·085%; the recovery of the gold was complete, the average current efficiency was 95·04%, and the nitric acid consumption 43·15%. During the electrolysis the free nitric acid content of the electrolyte fell from 3·38—3·70% to 0·80—0·97%, and the copper content increased from 0·69% to 2·39% per batch run of 1 kw.-hr. The original silver content of the electrolyte was 3·13—2·28%. A. A. ELDRIDGE.

Féry's new theory for the lead accumulator. E. DENINA (L'elettricista, 1926, **35**, 4; Chem. Abstr., 1926, **20**, 3135).—The observed facts are inadequately explained either by the classical double sulphation theory or by Féry's theory which, probably incorrectly, assumes the migration of lead from one plate to the other, including the discharge of lead ions at the cathode during discharge of the accumulator. In the prolonged electrolysis of sulphuric acid alone, or containing ammonium sulphate or benzenesulphonic acid, with lead electrodes, no lead was deposited at the cathode. (Cf. B., 1925, 997; 1926, 412). A. A. ELDRIDGE.

PATENTS.

Electrodeposition of metallic chromium. E. SUZUKI, Assr. to GENERAL ELECTRIC CO. (U.S.P. 1,600,076, 14.9.26. Appl., 31.7.25).—The metal is deposited from an electrolyte containing 5—10% of chromic acid, 5—15% of chromium sulphate, and 5% of boric acid, a lead anode being employed. M. COOK.

Electroplating [a non-metallic] article. J. R. BROWN and J. C. MULLINNIX, Assrs. to RELIANCE GAUGE COLUMN CO. (U.S.P. 1,599,608, 14.9.26. Appl., 11.2.22, renewed 21.6.24).—In electroplating a non-metallic article it is first coated with a cellulose derivative, such as celluloid, then covered with an electro-conductive material, and the desired metal finally deposited electrolytically upon it. M. COOK.

Applying non-corrosive coatings to metals. J. HÄRDÉN and H. T. TILLQUIST (E.P. 259,761, 13.10.25).—After coating with chromium electrolytically the metal is heated to a temperature not exceeding 1200° to expel any saline solution, moisture, or gas between the metal and the coating. After cooling to 500° the metal is subjected to a pressing process to promote alloying between the metal and the coating. M. COOK.

Electrolyte for electrolytic [rectifying] cells. C. C. CARPENTER, Assr. to WILLIARD STORAGE BATTERY CO. (U.S.P. 1,600,397, 21.9.26. Appl., 5.6.22).—The addition of citric acid to rectifying cells, in which the film-forming electrode is of aluminium, and the electrolyte is, for example, a mixture of ammonium and potassium phosphates, greatly increases the life of the cell. T. S. WHEELER.

Metallic filaments for electric incandescence lamps. NEUE GLÜHLAMPEN-GES.M.B.H., Assees. of M. HAUSCHILD (E.P. 259,810, 26.1.26. Conv., 19.11.25).

Accumulator plates. W. HADDON (E.P. 259,764, 16.10.25).

XII.—FATS; OILS; WAXES.

Fat from *Salvadora oleoides*. Khakan fat. C. K. PATEL, S. NARAYANA IYER, J. J. SUDBOROUGH, and H. E. WATSON (J. Indian Inst. Sci., 1926, **9A**, 117—132).—The seeds of *Salvadora oleoides* yield 42—43% of fat on exhaustive extraction with ether, whilst the native method of pressing gives 33%. The fat may be split by means of castor-seed lipase, 8% of castor seeds with 0·14% of acetic acid as activator completing the hydrolysis within 24 hours. The fat, which is yellowish-green in colour, and contains nitrogen and sulphur, is best refined by steam distillation to remove the odorous principle, followed by treatment with sodium hydroxide and fuller's earth, the fat then having the following characteristics: acid value, 0·38; iodine value, 14·4; Reichert–Polenske value, 5·3; saponif. value, 251·4; n_D^{60}, 1·4431; and Polenske value, 9·4. The fatty acids present as glycerides are caprylic, 4·4%; capric, 6·7%; lauric, 47·2%; myristic, 28·4%; oleic, 12·0%; and linoleic, 1·3%. The presence of stearic acid appears to be doubtful, the stearic acid detected in the hardened fat probably arising from the unsaturated acids originally present. The unsaponifiable matter contains sitosterol and *s*-dibenzylthiocarbamide, while the volatile oil, representing 1·5% of the expressed oil, is largely benzylthiocarbimide. Tables are given showing the analytical constants for fat obtained by various methods from new and old seeds, the results of splitting experiments, the relation between iodine value and refractive indices of hardened samples, and the composition of the fatty acids and derived esters. The fat is of the same type as palm-kernel and coconut oils, and although not edible, it is suggested as a useful ingredient for soap-making. E. HOLMES.

Oil from *Aleurites trisperma*. C. D. V. GEORGI (Malayan Agric. J., 1926, **14**, 290—291).—Nuts from *A. trisperma* consist of 56·4% of kernel containing 4·2% of moisture and 50·9% of oil (chloroform extract). The expressed oil, of yellow colour and sweetish odour, proves to be a semi-drying oil having the following characteristics: $d_4^{15.5}$ 0·935; iodine value, 111·4; saponif. value, 195·8; and acidity (as oleic acid), 1·1%. It is compared with the better-known candlenut oil. E. HOLMES.

Oil from *Aleurites* species. C. D. V. GEORGI (Malayan Agric. J., 1926, **14**, 292—295).—The shells and kernels of candlenuts may be satisfactorily separated by crushing the sun-dried nuts for a few minutes in an edge runner mill and then passing the mixture through a coarse (3 mm.) sieve. Oil expressed from the resulting material has iodine value 152·8 and acidity (as oleic acid) 2·4%. E. HOLMES.

Origin and detection of rancidity in fats and oils. J. PRITZKER and R. JUNGHUNZ (Z. Unters. Lebensm., 1926, **52**, 195—215).—The theory of autoxidation as the cause of rancidity is discussed. The Kreis test and that suggested by von Fellenberg were examined. Free fatty acids and fatty acid peroxides did not affect the Kreis reagent. Colour changes during the development of rancidity in a number of fats showed no regularity. Of many fatty acids and derivatives examined, only ethyl oleate gave a positive reaction with the Kreis reagent. The freshly-prepared compound was inactive, but after a few hours' exposure to light produced a slight coloration with the Kreis reagent, and after 14 days' exposure a very intense colour developed. At the same time, the ester gave a very definite peroxide reaction. Acetone did not affect the Kreis reagent. The theory of Powick (A., 1923, **ii**, 191; and B., 1924, 302) that acraldehyde peroxide is the active substance in the Kreis test is regarded as improbable. A. G. POLLARD.

Detection of fish oils. M. TSUJIMOTO (J. Soc. Chem. Ind. Japan, 1926, **29**, 561—564).—The method is based on the fact that in the determination of iodine value by Wijs' method, an insoluble precipitate is formed with fish oils, but not with vegetable oils. The test is best applied to the separated fatty acids. About 0·5 g. of fatty acids is dissolved in 10 c.c. of ether, 3—5 c.c. of a glacial acetic acid solution of iodine monochloride (about N) are added, and the solution is kept for two hours with frequent shaking at 15—20°. In the presence of fish-oil fatty acids a precipitate or turbidity is formed, whereas with fatty acids from vegetable oils the solution remains clear even after keeping. By this method it is found possible to detect about 1% of Japanese sardine oil or 5% of herring oil in linseed oil, or 0·2% of clupanodonic acid in linseed oil fatty acids. Analysis of the ether-insoluble iodochloro-additive compounds has proved them to be derived from the highly unsaturated acids of the series $C_nH_{2n-8}O_2$ and $C_nH_{2n-10}O_2$. The method is, therefore, suitable not only for the detection of fish oils, but also as a delicate test of the highly unsaturated acids, which commonly occur in fish and other fatty oils. K. KASHIMA.

PATENTS.

Improving the durability of liquid vegetable oils. H. BOLLMAN (E.P. 260,108, 9.11.25; cf. G.P. 337,169 and E.P. 160,840; B., 1921, 630 A; 1922, 945 A).—Liquid vegetable oils, such as sweet oil, salad oil, etc., are subjected to the usual refining and deodorising processes, and are then treated with 0·05—0·1% of lecithin, whereby their keeping properties are greatly improved. T. S. WHEELER.

Separating and obtaining the constituents of waste oils and fats. CONTINENTALE A.-G. F. CHEMIE, and R. TERN (E.P. 259,417, 1.12.25).—Waste oils etc. are subjected to multiple steam distillation *in vacuo* in three successive vessels at decreasing temperatures, a separation into saponifiable and unsaponifiable constituents being effected. The latter are removed from the distillation vessel as they separate, and may be recovered. The saponifiable portion is redistilled and dehydrated. If necessary a preliminary bleaching and desiccation may be undertaken, and the first distillation may be carried out in the presence of a concentrated solution of hydrogen peroxide. S. S. WOOLF.

Isolating oil, starch, albuminous substances, and cellulose from oleaginous materials. L. F. DAVID and G. FÉLIZAT (E.P. 245,100, 16.12.25. Conv., 29.12.24).—See F.P. 603,836; B., 1926, 987.

Oil presses with disintegrating means. H. ZANDER (E.P. 255,422, 18.5.26. Conv., 14.7.25).

Oil treating composition (U.S.P. 1,600,845).—See VII.

Revivifying spent [oil] filtering materials (U.S.P. 1,598,967).—See XVII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Electrometric determination of the acidity of writing inks. H. A. BROMLEY and A. DE WAELE (Analyst, 1926, **51**, 567—568).—The acidity of inks may be determined to an accuracy of 1% of p_H value for p_H 8 (beyond this degree of alkalinity the accuracy falls rapidly) by the use of the so-called quinhydrone electrode, which is not affected by the presence of charged colloidal particles. To 10 c.c. of the ink in a small beaker are added sufficient quinhydrone to saturate, and 5 c.c. of a standard buffer solution with a p_H of about 6 is placed in another beaker of the same size, together with 5 c.c. of distilled water and quinhydrone to saturate. After keeping for a few minutes the solutions are joined by a bridge of saturated potassium chloride solution. Each electrode consists of a spiral of stout platinum wire fused into the end of a short length of glass tubing having a small bulb containing mercury, and contact with the platinum is made by copper wires. A millivoltmeter is used having a third terminal, and the p_H value is found from the formula $p_H = (E/0{\cdot}0575) \pm K$ at 16°, where K is the known p_H of the buffer solution. D. G. HEWER.

Preparation of "treated oils," driers, etc. SEDLACZEK (Farben.-Ztg., 1926, **32**, 26—28, 81—82, 134—137).—A review of German patent literature. S. S. WOOLF.

Resin inclusions in bituminous coals. WIGGINTON.—See II.

PATENTS.

Manufacture of pigments. F. RAHTJEN and M. RAGG (E.P. 244,481, 11.12.25. Conv., 11.12.24).—Litharge, red lead, white lead, and other compounds of lead with inorganic oxyacids are converted into substances suitable for anti-fouling paints by heating them with high-boiling organic compounds, *e.g.*, vaseline, paraffin, ceresin, waste lubricant oils, waste anthracene and other hydrocarbons, and tar, pitch, or phenols, to about 300° in the absence of air. The lead oxides are

converted into a grey substance of extreme fineness. Such pigment, mixed with a non-reducing vehicle, neither thickens nor settles when used as an anti-corrosive paint. F. G. CROSSE

Process for producing lithopone. W. J. O'BRIEN. Assr. to GLIDDEN Co. (U.S.P. 1,600,772—3, 21.9.26, Appl., 26.9.24 and 16.11.25).—Zinc sulphate solution is acted on by barium sulphide solution in the presence of titanium dioxide, the precipitate so formed being treated as in the usual lithopone process. The resulting pigment has greater covering power and weather-resistance than ordinary lithopone. S. S. WOOLF.

Manufacture of phenol–formaldehyde resins. E. E. WALKER (E.P. 259,046, 9.4.25).—The condensation of 1 mol. of phenol with rather more than 1 mol. of formaldehyde in presence of alkaline condensing agents (*e.g.*, alkali sulphite, caustic alkali, etc.) is allowed to proceed to a stage short of precipitation of the resin, control being exercised by limitation of the viscosity of the reaction mixture. The resin is precipitated by the addition of sufficient acid to bring the mother-liquor to p_H 3·2—7 (the actual viscosity and p_H limits being determined for each specific reaction mixture). Resins so obtained are particularly light-coloured, permanent to light and air, and of high mechanical strength when hardened. S. S. WOOLF.

Improving steam-distilled wood turpentine. D. L. SHERK (U.S.P. 1,600,143, 14.9.26. Appl., 16.6.23). —Turpentine, obtained by treatment of pine wood with steam, is freed from all irritating odour, and rendered available as a complete substitute for gum turpentine, from the exudation of the pine tree, by agitation with 25% of a 20% solution of sodium hydroxide at 100—115° for 2 hrs. The product is distilled from the alkali with steam. An alcoholic solution of the alkali can also be employed. T. S. WHEELER.

Process of resinifying aliphatic aldehydes. W. O. HERRMANN and H. DEUTSCH, Assrs. to CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (U.S.P. 1,600,113, 14.9.26. Appl., 16.10.22. Conv., 20.10.21).—See E.P. 187,619; B., 1923, 730 A.

Manufacture of solvents [for varnishes] (E.P. 259,293).—See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Jelutong. B. J. EATON, C. D. V. GEORGI, and GUNN LAY TEIK (Malayan Agric. J., 1926, **14**, 275—285).—Jelutong, the resinous material derived from the latex of *Apocynaceæ Dyera*, is increasing in importance as a result of the American demand for this substance in the manufacture of chewing gum. The present authors describe a critical examination of the *Hevea* latex coagulants, acetic acid, alum, sulphuric acid and sodium silicofluoride as applied to the coagulation of *Apocynaceæ* latex of d_4^{23} 1·012—1·015. Each test is compared with a control coagulation conducted by dropping the latex into 1½ times its volume of alcohol, the dry jelutong being weighed. Sodium silicofluoride is an excellent coagulant, the maximum yield being obtained by the addition of 0·35 g. per litre of latex, but, owing to its toxic nature, its use is contrary to the United States food laws. Acetic acid, therefore, is recommended, the optimum yield being given when 0·5 g. of acid is used per litre of latex, the time of coagulation being about three days. Formaldehyde partially inhibits coagulation, and the substance obtained is hard and tough. It is suggested that evaporation of the latex by means of the spray process now used for rubber may prove satisfactory. E. HOLMES.

Adsorptive powders for rubber goods and a new theory of "blooming." R. DITMAR (Gummi-Ztg., 1926, **41**, 246—247).—Adsorptive powders, *e.g.*, "colloidal" clays, have a beneficial action on cold-vulcanised articles by adsorbing any residual sulphur chloride or solvent, and also any decomposition products of the former; they also check "after-vulcanisation" and impart smoothness. The "blooming" of hot-vulcanised rubber is attributed to the gradual liberation of hydrogen sulphide which, in contact with atmospheric moisture, reacts with sulphur dioxide produced by oxidation, with formation of sulphur. In the presence of an adsorptive powder these gases are adsorbed and react in the colloidal powder. D. F. TWISS.

Unsmoked sheet rubber prepared with dinitro-o-cresol. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1926, **8**, 536—537).—Dinitro-*o*-cresol as a mould preventive for rubber is as efficient as *p*-nitrophenol, and has no appreciable effect on the rate of vulcanisation. D. F. TWISS.

Comparison of smoked sheet rubber prepared with *p*-nitrophenol by the coagulating and soaking processes. H. P. STEVENS (Bull. Rubber Growers' Association, 1926, **8**, 538).

PATENTS.

Making rubberised fibrous compositions. W. G. O'BRIEN and P. BEEBE, Assrs. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,599,383, 7.9.26. Appl., 22.3.23).—Rubber is precipitated from a solution in a mixture of toluene and alcohol on to fibrous material; the toluene is removed subsequently by the action of superheated alcohol vapour. D. F. TWISS.

Manufacture of rubber. C. C. LOOMIS and H. E. STUMP, Assrs. to HEVEA CORP. (U.S.P. 1,599,282, 7.9.26. Appl., 10.4.23).—Rubber latex, by partial coagulation, is converted into a plastic paste, which is then formed into the desired shape and dried. D. F. TWISS.

Abrading materials [containing rubber] in the form of slabs etc. (E.P. 259,679).—See VIII.

XV.—LEATHER; GLUE.

Japanese dyeing tannins. III. Tannin in the leaves of *Rhus trichocarpa* (Yamahaze). Y. UEDA (J. Soc. Chem. Ind. Japan, 1926, **29**, 557—559. **IV. Leaves of *Rhus javanica* (Fushinoki).** Y. UEDA and K. ISHINADA (*ibid.*, 1926, **29**, 659—660).—III. The tannin $C_{20}H_{20}O_{14}$, extracted from *Rhus trichocarpa*, Mig., gave an octa-acetyl compound, m.p 159—160°. When hydrolysed with 50% sulphuric acid, gallic acid (about 60%) and hexose were produced. IV. The leaves of *Rhus javanica*, Lim., contain gallotannin (yield about 12·3%) and ellagic acid (about 1·8%). K. KASHIMA.

Extraction of chromium from chrome tanned leather with sodium potassium tartrate. II. N. I.

Berestovoj and L. Masner (J. Soc. Leather Trades Chem., 1926, **10**, 299—302; cf. B., 1925, 893).—Experiments on the influence of acidity on the extraction of chromium from chrome tanned leather by means of sodium potassium tartrate have shown that a minimum is obtained at p_H 3·58 which is not far from the isoelectric point of collagen p_H 4·7. The amount of chromium extracted increased with increasing amounts of sodium potassium tartrate solution, but decreased with increasing amounts of oxalic acid solution. Solutions of sodium hydroxide hydrolysed the leather, and the amount of chromium extracted was proportional to the amount of hydrolysis. D. Woodroffe.

Analysis of synthetic tanning materials. E. Wolesensky (U.S. Bur. Standards, Tech. Papers, 1926, **20**, 519—527).—The acidity is best measured by means of the potentiometer, but the titration may be made with N-sodium hydroxide solution, using Congo-red paper to determine the approximate end-point, and red and blue litmus papers for the final adjustment. Express the acidity in terms of sulphuric acid (A). For the total sulphur determination 3—8 c.c. of the diluted solution of synthetic tannin are filtered and evaporated nearly to dryness on a water bath with concentrated nitric acid. The residue is treated with further concentrated nitric acid, heated to boiling in a covered beaker as long as brown fumes are evolved, evaporated to 10—12 c.c., diluted with water, heated to near boiling, and the sulphates determined in the usual way. The result is expressed as sulphuric acid (T), and represents the total original sulphuric acid used in the sulphonation. To determine the total inorganic matter, a weighed sample of the material is evaporated to dryness in a weighed porcelain crucible, first on a water bath and then in an oven at 105—110° for 1—2 hrs., charred by gentle ignition, cooled, treated with 15 drops of concentrated sulphuric acid, heated gently at first, then to a moderate red heat, cooled, and the treatment with sulphuric acid and heat repeated until all carbonaceous matter has been completely oxidised. Cool, moisten with 2 drops of sulphuric acid, ignite gently, then more strongly to constant weight. The residue will be largely sodium sulphate, but it should be tested for other metals. Express result as sulphuric acid (S). If no soluble inorganic matter is present and $T < 2A$, the free sulphuric acid is $2A - T$, the acidity due to sulphonic acid is $T - A$ in terms of sulphuric acid, and the sulphuric acid combined as sulphonic acid is $2(T - A)$. If $T = 2A$, no free sulphuric acid is present and all the sulphonic acid is free. In presence of soluble inorganic matter, if $(T - S) = 2A$ no free sulphuric acid is present. If $(T - S) < 2A$ the free sulphuric acid is $2A + S - T$, combined sulphates is S, the acidity due to sulphonic acid (expressed as sulphuric acid) is $T - S - A$, and the amount of sulphuric acid combined as sulphonic groups is $2(T - S - A)$. If $(T - S) > 2A$ no free sulphuric acid is present, and part of the sulphonic acid is neutralised and is equal to $T - S - 2A$, expressed as sulphuric acid. The soluble sulphate is $2A + 2S - T$. For non-volatile matter, a sample is weighed out in a wide-mouthed weighing bottle, exactly neutralised with sodium hydroxide solution, shaken, and dried *in vacuo* over sulphuric acid. The last traces of moisture may be removed in a vacuum oven. Total organic matter is given by non-volatile matter less the inorganic matter and free sulphuric acid. For determining tanning material a sample of the syntan is mixed with barium acetate solution, containing a quantity of this salt equivalent to the amount of soluble sulphates and free sulphuric acid in the sample taken, diluted with water to 1 litre, left overnight, and the clear liquor used for the usual tannin analysis. The tanning material is the total non-volatile matter less the "non-tans" as found in the tannin analysis, the free sulphuric acid, and the difference between the weights of the soluble sulphates and of the acetates formed therefrom. D. Woodroffe.

Patents.

Treatment of hides. C. J. M. M. Le Petit, (E.P. [a] 250,907, 27.1.26, Conv., 18.4.25; [b] 253,549, 11.6.26, Conv., 12.6.25. Addn. to [a]).—(a) Ripe seeds rich in diastatic ferments are ground or crushed, de-oiled if necessary, and inoculated with pure or acclimatised lactic ferments, or other ferments, to produce a medium favourable to the culture of fungi. 1 pt. of potassium phosphate, magnesium chloride, calcium carbonate, zinc salts, or similar substances may be added per 1000 pts. of the fermented product, then inoculated with spores of *Aspergillus niger* or various species of *Penicillium* to produce ferments such as lipases, proteases, nucleases, and cytases. Hides are pasted on the hair side or on both sides with the product suspended in a warm and ventilated medium at a temperature of 25—38° and unhaired. The action of the product may be completed by certain enzymes such as papain, bromeline, etc. (b) Bran or some other culture medium is treated with a 5% solution of lactic acid tanning extract, particularly quebracho extract, or substance capable of producing selective sterilisation, sown with *Aspergillus oryzæ*, and the product used for unhairing hides. D. Woodroffe.

Removing hair from green hides. M. Bergmann, E.I. and A. Immendörfer (U.S.P. 1,599,358, 21.9.26. Appl., 13.9.24).—Hides are immersed in a solution of an alkali sulphide to which has been added at least an equimolecular quantity of an ammonium salt and also a soluble silicate. D. Woodroffe.

Manufacture of shaped articles from artificial horn made of casein. E. C. R. Marks. From Int. Galalith-Ges. Hoff & Co. (E.P. 259,418, 1.12.25).—Relatively dry, unhardened or slightly hardened casein containing about 20% of water is shaped, and may or may not be given a preliminary hardening with solutions of aluminium salts or other metal salts having a hardening action, *e.g.*, tannic acids or hexamethylenetetramine, softened preferably by the action of heat, then shaped and dried. D. Woodroffe.

Coloured patent leather and the process of making the same. Snyder–Welch Process Corp. (E.P. 255,022, 15.3.26. Conv., 8.7.25).

Production of decorative leather or like pliable skin goods. F. Zimmer, and Lederwerke M. Zimmer A.-G. (E.P. 260,122, 9.12.25).

Protecting materials from mildew (E.P. 259,690).—See V.

XVI.—AGRICULTURE.

Determination of manurial requirements of soils by means of plant and soil analyses. W. Lange (Z. Pflanz. Düng., 1925, **A6**, 193—228).—The manurial requirements of six soils of different types, as determined by vegetation experiments, are compared with the indications given by the results of detailed analyses by various methods of the soils and the plants grown on them. The amounts of phosphoric acid and potassium extractable by hydrochloric acid, and the molecular proportions of silica, alumina, and total bases, as suggested by Ganssen, failed to give useful indications of the fertiliser requirements. The Neubauer seedling method was more satisfactory, but distinguished only between soils having pronounced shortage or abundance of available nutrients. Lemmermann's method (relative solubility of P_2O_5 in 1% citric acid) gave results in general agreement with the vegetation experiments.
C. T. Gimingham.

Neubauer's method for determination of available phosphoric acid and potassium [in soils]. H. Hähne (Z. Pflanz. Düng., 1925, **A6**, 238—248).—Various factors which may affect the results obtained by Neubauer's seedling method for determination of fertiliser requirements of soils were investigated. The amounts of P_2O_5 and K_2O taken up by the rye seedlings depend to some extent on the type of seed, and are markedly influenced by the temperature at which the plants are grown. Variations in intensity of illumination are less important. The results differ according to whether the control figure allowed for is the content of P_2O_5 and K_2O in the seeds or in plants grown in pure sand. C. T. Gimingham.

Determination of the fertiliser requirements of soils by the Neubauer method. H. Kruppa (Z. Pflanz. Düng., 1926, **A7**, 73—127; cf. Neubauer B., 1925, 220).—A critical study of the seedling method of Neubauer (Z. Pflanz. Düng., 1923, **A2**, 229) for the determination of the plant nutrient requirements of soils. The method, whilst capable of revealing large differences in the supply of available plant nutrients in soils, cannot afford a quantitative basis for determining fertiliser requirements. The results obtained are influenced by the water content of the soil, by illumination, and by other conditions of experiment. The deductions from data obtained by the method are not necessarily applicable to field conditions, where the factors affecting growth, other than plant food supply, are widely different from those obtaining in laboratory experiments. G. W. Robinson.

Decomposition of raw phosphate with peat. S. Rozanov (Nauch. Agron. Zhur., 1925, **2**, 110—120; Chem. Abstr., 1926, **20**, 3325).—Determinations were made of the solubility of the phosphate (in raw phosphate) in mixtures with peat (1 : 8—16), 1—180 days after composting. For six months the solubility was less in citrate solution than in water. The water-soluble phosphate decreased with time from 9 to 7·3% for the 1 : 8 compost, and from 15·1 to 14·5% for the 1 : 16 compost. When water-soluble phosphate, in the form of calcium phosphate, was mixed with peat it was partly adsorbed, and could not be completely removed by solvents. A. A. Eldridge.

Solubility and rapidity of action of different calcareous dressings. E. Manshard (Z. Pflanz. Düng., 1926, **A7**, 31—53).—The author has studied the solubility and effectiveness of dolomitic limestone, chalk marl, and soft chalk in ammonium chloride solution. Whilst in the case of the first two substances the rate of solution and solubility were greatly affected by the degree of division, in the soft chalk the coarser material (0·30—0·75 mm.) was practically as soluble as the finer material (< 0·15 mm.). This is attributed to its porous structure. In considering the value of such dressings, it is therefore necessary to take into account not only chemical composition and fineness of grinding, but also the fine structure of the material. The effects of different dressings on soil acidity were in agreement with the laboratory data. During the period of the experiments (two years) there was no appreciable effect on the acidity of the soil below the zone in which the dressings were incorporated. "Endlaugenkalk," a by-product from potash works, containing water 37·64%, chlorine 8·51%, magnesia 8·78%, total lime 25·99%, and calcium oxide 13·68%, showed a comparatively low efficiency as a soil dressing. G. W. Robinson.

Alkali formation in soils. J. Zink (Z. Pflanz. Düng., 1925, **A6**, 229—237).—The interactions of calcium carbonate and bicarbonate with alkali salts in presence and absence of carbon dioxide are discussed, with special reference to the work of Münter (cf. Z. Pflanz. Düng., 1923, **B2**, 289). Evidence is brought forward that in the soils of humid regions neither the interaction of calcium carbonate and alkali salts nor the hydrolysis of calcium carbonate can account for any appreciable degree of alkalinity. C. T. Gimingham.

Tolerance of plants for sodium chloride. C. B. Lipman, A. R. Davis, and E. S. West (Soil Sci., 1926, **22**, 303—332).—Wheat, barley, and peas grown in solution cultures all show a high resistance to sodium chloride, and, under certain conditions, sodium chloride is stimulating to wheat at concentrations as high as 4000 p.p.m. Peas are less resistant than wheat. Some growth is made by these plants with 10,000—15,000 p.p.m. of sodium chloride present, but none at higher concentrations. The most marked depression of growth occurs at about 8000 p.p.m. The results varied greatly, according to the time of year at which the plants were grown; climatic conditions are important in determining tolerance for sodium chloride. C. T. Gimingham.

Effect of flooding with sea-water on the fertility of the soil. H. J. Page and W. Williams (J. Agric. Sci., 1926, **16**, 551—573).—The harmful effects consequent on flooding of agricultural land by sea-water are attributable to the partial replacement of exchangeable calcium by sodium. Treatment of flooded land with lime or gypsum did not produce a noticeable improvement in tilth within 12 months, although the expected chemical action had taken place. The most satisfactory

method of dealing with such land appears to be the establishment of a ley of lucerne, clover, or "seeds."
G. W. ROBINSON.

Why are serpentine and other magnesian soils infertile? A. GORDON and C. B. LIPMAN (Soil Sci., 1926, **22**, 291—302).—Water extracts of soils derived from serpentine and other rocks with a high content of magnesium are characterised by very low concentrations of nutrient ions and an alkaline reaction (p_H generally about 8·1). The ratio of the concentration of magnesium to the total concentration of the soil extract is not necessarily high. The growth of barley in the soil extracts is poor, but addition of essential ions and a lowering of the p_H enabled good growth to take place. The infertility of these soils is not due to a too high content of soluble magnesium, but to a high p_H and a deficiency in certain ions, chiefly phosphate and nitrate.
C. T. GIMINGHAM.

Colloidal silica and silicates in agriculture. C. GRANVIGNE (Rev. gén. Colloïd., 1926, **4**, 193—199, 229—234).—A summary is given of the literature concerning the part played by colloidal silicates in plant nutrition and soil amelioration. The value of colloidal silicates in retaining fertiliser salts and reserve bases in the soil is pointed out. Experimental work indicating the action of silicates in increasing the availability of soil phosphates is cited. A. G. POLLARD.

Rôle of aluminium in soil infertility and toxicity. F. HARDY (J. Agric. Sci., 1926, **16**, 616—631).—A theoretical discussion of the problem of the occurrence of "active" aluminium in soils. The view that aluminium in a state toxic to plants does not occur when the reaction of the soil or nutrient medium lies between p_H 4·7 and p_H 8·5 is criticised. Aluminium may possibly enter plant roots and, under certain conditions, be translocated as simple ions, complex colloidal electrolytes, co-ordinated complex anions, or organic aluminium compounds. The true toxic effect is only exerted by simple ions or the more soluble colloidal electrolytes. Non-toxic forms of aluminium may accumulate in certain tissues and render plants liable to certain diseases. G. W. ROBINSON.

Wood ashes as an ameliorant of soil acidity. S. K. MITRA and L. N. PHUKAN (Agric. J. India, 1926, **21**, 357—365).—Field experiments, continued for a number of years with several crops, show that applications of wood ashes on certain acid and infertile soils in Assam enable good crops to be grown by reducing the acidity and supplying needed plant food. The use of wood ashes, together with dung, gave the best results.
C. T. GIMINGHAM.

Injurious action [on soils] of large applications of lime. E. W. BOBKO, B. A. GOLUBEV, and A. F. TÜLIN (Z. Pflanz. Düng., 1925, **A6**, 128—168).—Excessive applications of lime act injuriously (*i.e.*, depress crop yields in vegetation experiments) not only on peat soils, but also on mineral soils, especially light uncultivated forest soils. Biological processes are stimulated and large amounts of bicarbonates, ammonia, nitrates, and sometimes nitrites accumulate in the soil solution. The harmful action is attributed to the excessive amounts of ammonia produced; it can be prevented by leaching the soil. No such injurious action follows large dressings of lime on heavy soils, and there is no accumulation of ammonia. The explanation given did not hold in the case of an experiment with vetches in a garden soil.
C. T. GIMINGHAM.

Variability of the effect constant ("Wirkungsfaktor") in growth curves. A. RIPPEL (Z. Pflanz. Düng., 1926, **A7**, 1—12, 16). **Constancy of the effect constant in growth curves.** E. A. MITSCHERLICH (*Ibid.*, 13—15).—Polemical. Rippel attempts to show from data already published by Mitscherlich that under certain conditions the effect constant may vary inversely with the absolute yield. Mitscherlich criticises Rippel's conclusions on the ground of the unsuitability of the data and also on statistical grounds.
G. W. ROBINSON.

Experimental proof of the incorrectness of the Mitscherlich–Baule law connecting yield with growth factors. A. RIPPEL (Z. Pflanz. Düng., 1926, **A8**, 65—80).—In pot experiments with oats, it is shown that the Mitscherlich effect constant ("Wirkungsfaktor") is about twice as great in a series with large dressings of nitrogen as ammonium nitrate, as in another series with smaller dressings of nitrogen. The experiments are held to disprove the constancy of the Mitscherlich factor.
G. W. ROBINSON.

Acidity of moor soils. I. Determination of total acidity by the Tacke–Süchting method. B. TACKE and T. ARND (Z. Pflanz. Düng., 1925, **A6**, 295—306).—Consideration of the conditions in moor soils and experimental comparison of the Tacke–Süchting method with other methods for the determination of soil acidity, leads to the conclusion that the Tacke–Süchting method gives the most reliable figures for the total acidity of moor soils. C. T. GIMINGHAM.

Quinhydrone method [for determination of reaction of soils] with reference to different forms of soil acidity. H. KAPPEN and R. W. BELING (Z. Pflanz. Düng., 1925, **A6**, 1—26).—Use of the quinhydrone electrode is recommended for determination of the p_H of soils, and the bearing of the results obtained on the recognition of different forms of soil acidity is discussed. There is no recognisable relation between the p_H of suspensions of soil in *N*-potassium chloride solution and in water, because the figures are influenced to different extents by exchange acidity, hydrolytic acidity, and acidity due to neutral salt decomposition. Suspensions of acid soils, in both water and *N*-potassium chloride solution, show a lower p_H than the corresponding filtrates. Humic acid, prepared from sugar, is more suitable for the study of "neutral salt decomposition" acidity with the quinhydrone electrode than hydrated manganese dioxide (cf. Hümmelchen and Kappen, A., 1925, i, 218). C. T. GIMINGHAM.

Determination of nitrate in soils by means of the diphenylamine reaction. H. RIEHM (Z. Pflanz. Düng., 1926, **A7**, 22—30).—The extract obtained by shaking 25 g. of moist soil with 50 c.c. of sodium chloride solution (40 c.c. of saturated solution per litre) is tested colorimetrically for nitrite and nitrate by means of the

following reagents. For nitrite, 60 mg. of diphenylamine and 325 c.c. of dilute sulphuric acid (1 : 3) made up to 500 c.c. with strong sulphuric acid; for nitrate, 170 mg. of diphenylamine and 330 c.c. of water made up to 1000 c.c. with strong sulphuric acid. In the absence of nitrites 2 c.c. of the extract are mixed with 5 c.c. of the nitrate reagent, and the mixture cooled and compared colorimetrically with a standard nitrate solution. Where nitrites are present a preliminary determination is made with the nitrite reagent. G. W. Robinson.

Direct effect upon plant cells of the hydrogen-ion concentration of the nutrient medium. W. Mevius (Z. Pflanz. Düng., 1925, **A6**, 89—98).—A review of the literature on the subject. C. T. Gimingham.

Determination of absorptive capacity and degree of unsaturation of soils. E. W. Bobko and D. L. Askinasi (Z. Pflanz. Düng., 1925, **A6**, 99—127).—The absorptive capacity of a soil is determined by saturating the soil with a barium compound, extracting with hydrochloric acid and determining the barium as sulphate. The procedure must be modified if carbonate is present. If a soil is saturated with hydrogen ions and treated with barium chloride, the amount of acid in the extract corresponds to the total absorptive capacity as determined by the barium method. Treatment alternately with barium chloride and 0·05 N-hydrochloric acid decreases the absorptive capacity of highly absorptive soils, such as chernozem, probably by destroying the absorbing complex. Podsol soils show no change under this treatment. Iron is absorbed by soils from a 0·05N-hydrochloric acid solution, but such absorption of iron does not affect the total absorptive capacity. By subsequent treatment with barium chloride, part of the iron goes into solution; the remainder does not seem to be held in the absorbing complex. C. T. Gimingham.

Measurement of concentration of carbon dioxide in the air close to agricultural crops. H. J. Keuhl (Z. Pflanz. Düng., 1925, **A6**, 321—378).—A detailed account is given of a large number of determinations of carbon dioxide in the air in the neighbourhood of various crops growing on two different soils and with different manurial treatments. Determinations were made close to the soil, at the level of the lower leaves, at the level of the upper leaves, and above the plants. The concentration of carbon dioxide in the air is affected by the meteorological conditions, by the intensity of assimilation, and by the condition of the soil and the manuring. Dung alone appeared to reduce the concentration, mineral fertilisers alone or with dung increased it. Further, the amount of carbon dioxide evolved from the soil was shown to be insufficient for the needs of the plants, and the amount present in the air about the plants is dependent on the density of the vegetation. There is a marked shortage of carbon dioxide where plants are grown very close together. There is thus evidence in favour of the possibility of a practical value from the use of "carbon dioxide manures," but further work on this subject is necessary. The results are in general agreement with those of Lundegardh ("Der Kreislauf der Kohlensäure in der Natur," Jena, 1924). C. T. Gimingham.

Decomposition of ammonium thiocyanate into carbon disulphide and mellon, and decomposition of mellon into ammonia and carbon dioxide. Gluud, Keller, and Klempt.—See VII.

Varying characteristics of three types of wheat grown under the influence of identical environment. Herman.—See XIX.

Patents.

Treatment of growing plants. [Fungicide.] I. H. H. Jacobsen (E.P. 259,803, 24.12.25).—Pulverised copper sulphate (7—8 pts.) is mixed with burnt gypsum (100 pts.) and the requisite quantity of lime for neutralisation, and is treated with water or a solution of copper sulphate (25 pts.). Much heat is evolved, the copper sulphate goes into solution, and the water present is then absorbed by the gypsum as water of crystallisation. The whole sets to a solid mass in which the copper sulphate is uniformly disseminated. The product is pulverised, and, when strewn on growing plants, has no deleterious action, and inhibits fungoid growths. T. S. Wheeler.

[Mixed] fertiliser corrective. F. W. Freise, Assr. to American Cyanamid Co. (U.S.P. 1,599,226, 7.9.26. Appl., 23.7.23).—100 lb. of ammonium sulphate are added to 1 ton of mixed fertiliser, obtained by the action of sulphuric acid on a mixture of phosphate rock and calcium cyanamide, while it is still moist after preparation. The mass rapidly becomes dry and granular, and the percentage of available phosphate is increased. T. S. Wheeler.

Drying of agricultural or horticultural products such as hops, seeds, or the like. V. Elkington (E.P. 259,270, 6.6.25 and 14.8.25).

Compounds of copper for use in combating fungi (U.S.P. 1,598,982).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Causes of errors in saccharimetric analysis. M. S. Filosofov (Zapiski [Russia], 1926, **3**, 185—199; Chem. Abstr., 1926, **20**, 3094).—Twelve sources of error in industrial saccharimetric analysis are enumerated and assessed. The total error may be 0·48°. A. A. Eldridge.

Behaviour of different starches towards dyestuffs and iodine [and bromine]. II. J. Huebner and K. Venkataraman (J. Soc. Dyers and Col., 1926, **42**, 327—332).—Prolonged (24 hrs.) action of iodine vapour on six starches affords the following colours: maize, bluish-black; potato, brownish-khaki; rice, dull violet, deeper and browner than wheat (dull violet); sago, mole colour; tapioca, faint drab. The same colours are obtained much more rapidly by grinding the starch (3 g.) with iodine (0·5 g.). The dried gelatinised starches are affected much less rapidly. In the presence of water the colour change is much more rapid, and in 24 hrs. all the starches have become black. Cold benzene does not extract iodine from maize starch; hot extraction affords a starch drying to a pale violet colour. Heating the original coloured maize starch at 100° for 12 hrs. does not entirely destroy the colour. On addition of water

the coloured maize, wheat, and rice starches are unchanged, potato and tapioca starches turn blue, and sago bluish-violet. The amount of iodine vapour absorbed by the starches is roughly proportional to their ash alkalinity. Pastes made from the original starches with cold water rapidly turn blue in contact with iodine vapour, whilst the dry starches and starch pastes do not. From aqueous solutions of iodine in potassium iodide the different starches absorb varying amounts of iodine, the differences being, however, much smaller than either the corresponding differences in the size of the granules or than the differences found (B., 1926, 436) in the absorption of dyestuffs by different starches. These observations and the fact that the raw starch absorbs in each case less iodine than the dried gelatinised preparations under similar conditions indicate that the absorption of iodine by starches is a chemical phenomenon. The order of the starches is, with slight exception, the same when arranged according to iodine absorption as when according to granule size, and similar absorption curves from solutions of iodine of different strengths were obtained in all cases. The absorption of iodine from solutions in organic solvents such as benzene, whilst small in amount, shows greater differences among the starches examined, rice starch absorbing 10 times as much iodine from benzene solution as sago or tapioca starch. Of the different solvents, alcohol gives the smallest absorption, and benzene slightly greater. Absorption from chloroform is nearly twice as great as from benzene. A little over 3 g. of potato starch completely exhausts 100 c.c. of saturated aqueous iodine. The organic solvents do not afford the characteristic blue colour. The absorption of iodine by starch from mixtures of alcohol and water is normal as compared with the absorption of dyestuffs by starch under similar conditions (*loc. cit.*). With potato starch minimum absorption occurs with 99% alcohol. Identification of the constituent starches in a mixture by means of the microscope is simplified by dyeing the mixture with a basic dyestuff, without mordant. The different starches are dyed different shades, and in a uniform mixture it is possible to calculate roughly the percentage of each starch by counting the number of different granules in the microscopic field. Exposure to bromine vapour affords similar differences in colour, the colours obtained by 72 hrs.' exposure being: maize, ochre; potato, pale yellow; rice, light buff; sago, pale yellow; tapioca, cream; wheat, ochre. As with iodine, the colours are unaffected by subsequent exposure to air.

R. BRIGHTMAN.

PATENT.

Revivifying spent [sugar] filtering materials. S. HILLER, Assr. to S. HILLER, INC. (U.S.P. 1,598,967, 7.9.26. Appl., 21.12.23).—Kieselguhr, fuller's earth, or other spent filtering material from the sugar or oil industry is passed through two rotating kilns in series, in the first of which it is preheated and dried by hot gases from the second kiln, in which it comes into contact with a jet of burning oil, which initiates combustion of the carbonaceous material retained in the pores of the spent substance. Air is admitted to complete the combustion.

T. S. WHEELER.

XVIII.—FERMENTATION INDUSTRIES.

PATENTS.

Fermentation processes for *n*-butyl alcohol and acetone [from artichokes]. A. P. H. DESBOROUGH, A. C. THAYSEN and B. M. GREEN (E.P. 259,631, 6.5.25).—A mash containing 50% of the wet tubers of Jerusalem artichokes (*Helianthus tuberosus*) is boiled for 2 hrs. at a pressure of 1·7 atm. with 0·2% of sulphuric acid to invert and render fermentable the carbohydrates present. Chalk is added to neutralise free acid, and after addition of water to reduce the concentration of the mash to 20—30% calculated on fresh tubers, fermentation is induced by adding 1—10% of a culture of an acetone bacterium and keeping the mash at 37—39° for 25—65 hrs. A yield of 12 gallons of a crude mixture of *n*-butyl alcohol and acetone per ton of fresh tubers is obtained.

T. S. WHEELER.

[Fermentation] treatment of fruits, vegetables, etc. (E.P. 259,625 and 259,895).—See XIX.

Malt-kilns. P. REISSHAUER (E.P. 259,473, 31.3.26).

XIX.—FOODS.

Varying characteristics of three types of wheat grown under the influence of identical environment. R. S. HERMAN (Cereal Chem., 1926, 3, 244—252; Chem. Abstr., 1926, **20**, 3320).—Kharkov, Blackhull, and Kanred wheats varied widely in baking characteristics, but not in milling qualities; variations in ash and protein content, absorption, p_H, and viscosity were evident. The protein content gave no indication of the fermentation period.

A. A. ELDRIDGE.

Comparison of the official method of ashing plant tissues and products with the Hertwig and Bailey method. C. F. ROGERS (Cereal Chem., 1926, **3**, 226—232; Chem. Abstr., 1926, **20**, 3318).—The difference in accuracy between the two methods is small; substances which are rich in fat give a higher apparent ash content by the latter method.

A. A. ELDRIDGE.

Commercial protein test on wheat and some of its problems. W. O. WHITCOMB and J. P. LEWIS (Cereal Chem., 1926, **3**, 232—243; Chem. Abstr., 1926, **20**, 3321).—Variation of moisture content is important in the commercial protein test, but the difference in protein between scoured and unscoured portions of smutty wheat is small (0·11%). Pure smut balls gave a protein value of 18·00%, 1·53 times as great as that of the wheat. Control samples of wheat and flour showed a maximum variation of 0·21% and 0·18% respectively.

A. A. ELDRIDGE.

Oxygen-acetate method of ash determination in flour. G. L. BRENDEL (Cereal Chem., 1926, **3**, 222—226; Chem. Abstr., 1926, **20**, 3320).—3 g. of the sample are charred, 2 c.c. of $N/4{\cdot}2$ calcium acetate solution added to the warm cinder, which is then dried, and the mass is ignited at 815°, oxygen being delivered into the muffle for 10—15 min. after an interval of 3—4 min. Low platinum or porcelain, but not silica, crucibles are satisfactory. A blank determination is performed with the calcium acetate solution.

A. A. ELDRIDGE.

Determination of acids in acid foods. W. U. Behrens (Z. angew. Chem., 1926, **39**, 1350—1351).—The aqueous extract of the foodstuff is acidified with a few drops of sulphuric acid and extracted with ether. The ethereal solution is titrated with standard alkali using phenolphthalein as indicator to obtain the total acidity (free and combined). The titrated solution is treated with an excess of alkali, and the ether is distilled off. The residual solution is acidified with phosphoric acid and distilled in steam to separate the volatile acids (acetic, butyric, etc.) from the non-volatile (lactic) acid. The distillate is titrated as before, and the difference between the two results is reckoned as non-volatile acidity. A second portion of the aqueous extract of the food is used for the determination of the hydrogen-ion concentration. The content of free acid in the original food may then be calculated from the equation: (free acidity)/(total acidity) = $H^{\cdot}/(H^{\cdot} + K)$, where $H^{\cdot}$ is the hydrogen-ion conductivity and K is the dissociation constant of the acid. If more than one acid is present the proportions of each may be found by the ether–water division method (Behrens, A., 1926, 1266). A. R. Powell.

Detection of incipient putrefaction in meat. R. Herzner and O. Mann (Z. Unters. Lebensm., 1926, **52**, 215—242).—Detailed analysis of numerous samples of meat, stored under various conditions, indicated that changes in the ratio, pure protein nitrogen : total nitrogen, were coincident with the early stages of putrefaction. Determination of the hydrogen-ion concentration of the meat juice itself, or of an aqueous extract of meat, also affords a means of detecting the commencement of putrefaction in the flesh of warm-blooded animals; p_H figures of 6·0—6·2 mark the initial stages, and figures above 6·2 were obtained with meat which was definitely decomposing. Preservation of meat with antiseptics of marked basic or acidic properties precludes the use of this method. A. G. Pollard.

Composition of the "frothy layer" in honey. J. Fiehe (Z. Unters. Lebensm., 1926, **52**, 242—244).—During the storage of extracted honey minute gas bubbles rise to the surface, and ultimately form a frothy layer. Frothing of candied honey was induced by warming. Analyses of the froth-layer indicated a difference from normal honey. The lævulose content increased and the dextrose decreased to a corresponding extent. Neither alcohol nor carbon dioxide was detected, and no fermentative action could be discerned. Froth formation occurred when a current of air was drawn through honey, and it is considered that frothing in stored honey is merely the effect of escaping air dissolved by the honey during the process of extraction.
A. G. Pollard.

Physical and chemical characteristics of maturing apples as related to the time of harvest. J. R. Neller and F. L. Overley (Wash. Agric. Exp. Sta. Bull., 1926, [205], 1—36; Chem. Abstr., 1926, **20**, 3310).—For the two varieties examined, the best harvest dates are those at which the total sugar ceases to increase rapidly; all samples reached the same sugar content in storage. Formulæ are devised to correlate the physical and chemical data for the apples. A. A. Eldridge.

Determination of the density of milk. J. Gerum (Z. Unters. Lebensm., 1926, **52**, 268—269).—To avoid the excessive washing of apparatus in routine work, the density is determined in a glass tube 22 cm. long and 4·0—4·2 cm. diam., having a wide-bored glass stopcock and delivery tube at the lower end. Hydrometer and thermometer remain in the tube throughout, each sample of milk being drawn off from below. A. G. Pollard.

Proximate composition of beef. C. Chatfield (U.S. Dep. Agric. Circ., 1926, [389], 1—18; Chem. Abstr., 1926, **20**, 3198).

Patents.

Yeast assistant [for making bread]. A. H. Fiske, Assr. to Rumford Chem. Works (U.S.P. 1,599,563, 14.9.26. Appl., 3.6.25).—Potassium nitrate, or other non-deliquescent nitrate, *e.g.*, sodium or ammonium nitrate (1 pt.), is added to a mixture of sodium chloride (25 pts.), calcium sulphate (25 pts.), calcium phosphate (10 pts.), ammonium chloride (10 pts.), and flour (29 pts.), and 1 pt. of the product is mixed with about 200 pts. of flour for bread making. The growth of yeast in the bread is greatly improved. T. S. Wheeler.

Treatment of fruits, vegetables, and residues therefrom. W. Matzka (E.P. 259,625, 8.4.25).—The material is disintegrated, extracted with water, and the insoluble residue separated. The weak aqueous extract obtained is fermented, with regular adjustment of acid content and addition of sugar characteristic of the raw material, by the aid of a culture which has been propagated upon portions of the liquor to be treated (cf. E.P. 259,895, following), and consequently will not destroy the natural aroma of the raw material. The fermented extract when filtered and clarified may be used as a beverage before or after distillation, whilst the residue is useful as cattle food. F. R. Ennos.

Fermentation treatment of fruits, vegetables, etc. W. Matzka (E.P. 259,895, 8.4.25).—A culture for the fermentation of aqueous extracts from fruit or vegetable residues is prepared by inoculating a portion of the extract in question, containing about 12% by volume of alcohol, with the ferment and propagating it with regulation of the acidity and addition of quantities of sugar characteristic of the raw material. The ferment is filtered, washed with water, and shaken with an aqueous solution containing 12% of alcohol and small quantities of tartaric acid and glycerin to stabilise it. It is again filtered and washed with an aqueous solution of glycerin and finally with water, after which it can be preserved with exclusion of air or used at once. F. R. Ennos.

Separating proteins and other matter from whey in soluble form.—R. W. Bell (U.S.P. 1,600,161, 14.9.26. Appl., 15.4.26).—After removal of the casein and fat, the whey has its acidity adjusted to p_H 7·0 by addition of a suitable alkali, and is then heated to 60°. It is evaporated, preferably in a vacuum pan, at a temperature below that at which the albumin coagulates until the lactose just fails to crystallise, and is then cooled and maintained at 0° until the maximum crystallisation of lactose has occurred. After removal of the lactose crystals by a centrifuge and reduction of the salts by electrodialysis, the liquid is adjusted in acidity

to p_H 7·3 and evaporated below the coagulating temperature of the albumin. The resulting product is a powder soluble in water, and contains practically all the proteins, part of the salts, and a small part of the lactose of the whey. F. R. Ennos.

Method for isolating the aromatic principle contained in roasted coffee. Internat. Nahrungs- und Genussmittel A.-G. (E.P. 246,454, 4.1.26. Conv., 23.1.25).—Roasted coffee is heated in a high vacuum to a suitable temperature, for example under 2—5 mm. at 100—110°. The aromatic constituents volatilise with the water contained in the coffee, and are condensed, first in receivers at 0°, then in others at — 20° to — 80°, and finally in receivers cooled by liquid air at — 190°, where the most volatile constituents, such as diacetyl and other aliphatic α-diketones, acetaldehyde and methylethylacetaldehyde, are condensed. To assist the expulsion of the aromatic substances a little steam or water may be supplied during the distillation, or 1—5% of water may be added previously to the coffee. The products are separated from water by extraction with organic solvents or by absorption in fats or oils, acids such as acetic acid being removed by repeated shaking with water or carbonate solution. Alternatively, the condensate is subjected to repeated steam distillation in a vacuum, followed by separation with organic solvents as before. The yellow or brown oil which is obtained is used for flavouring foodstuffs. A. Davidson.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Essential oil from the wood of *Erythroxylon monogynum*, Roxb. B. S. Rao, V. P. Shintre, and J. L. Simonsen (J. Indian Inst. Sci., 1926, **9A**, 145—148).—Steam distillation of the wood of *E. monogynum* from Mysore yields 1·15% of a viscous reddish-brown oil having d_{30}^{30} 0·9499, n_D^{30} 1·4998, $[\alpha]_D^{30}$ —43·9°, acid value 5·7, ester value 22·7, ester value after acetylation 74·7, and consisting of a mixture of sequiterpenes and sesquiterpene alcohols. The sesquiterpene fraction contains bisabolene and possibly cadinene. No indication of the presence of the crystalline alcohol, $C_{20}H_{32}O$, described by Schimmel & Co. (cf. B., 1905, 679) could be found in the sesquiterpene alcohols.

E. H. Sharples.

Essential oil from the rhizomes of *Curcuma aromatica*, Salisb. B. S. Rao, V. P. Shintre, and J. L. Simonsen (J. Indian Inst. Sci., 1926, **9A**, 140—144).—Steam-distillation of the disintegrated roots yields 6·1% of a greenish-brown oil (d_{30}^{30} 0·9139, n_D^{30} 1·5001, $[\alpha]_D^{30}$ — 12·5°, acid value 0·9, ester value 2·03, ester value after acetylation 58·66) having the following approximate composition: *d*-camphene, 0·8%; *d*-camphor, 2·5%; sesquiterpenes, 65·5%; sesquiterpene alcohols, 22·0%; acids (caprylic and *p*-methoxycinnamic), 0·7%; unidentified, 8·5%. The sesquiterpene fraction consists mainly of l-*curcumene*, b.p. 127—129°/6 mm., d_{30}^{30} 0·8760, n_D^{30} 1·4929, $[\alpha]_D^{30}$—21·5°, $[R_L]_D$ 67·67, (*trihydrochloride*, m.p. 84—85°; *trihydrobromide*, m.p. 73—74°; *nitrosate*, m.p. 100·4°), which, when dissolved in acetic anhydride and treated with one drop of sulphuric acid, gives a bright pink colour which becomes dark red on warming. When treated with sulphur according to Vesterberg's method, *l*-curcumene forms no naphthalene derivative.

E. H. Sharples.

Essential oil from the rhizomes of *Kaempferia galanga*. P. M. B. Panicker, B. S. Rao, and J. L. Simonsen (J. Indian Inst. Sci., 1926, **9A**, 133—139).—Prolonged steam-distillation of the roots of *K. galanga* yields 2·4—3·88% of oil (calculated on the dry material) containing ethyl *p*-methoxycinnamate (20—40%) and ethyl cinnamate (cf. Hariharan and Sudborough, A., 1925, i, 1149). The ester-free oil (d_{30}^{30} 0·8792—0·8914, n_D^{30} 1·4773—1·4855, $[\alpha]_D^{30}$ —2·6° to —4·5°, acid value 0·5—1·3, saponification value 99·7—109·0, saponification value after acetylation 110·1—116·3) consists mainly of *l*-Δ^3-carene (b.p. 166—167°/685 mm., d_{30}^{30} 0·8606, n_D^{30} 1·4684, $[\alpha]_D^{30}$ —5·72°) and *p*-methoxystyrene (possibly formed by decomposition of ethyl *p*-methoxycinnamate) with small quantities of camphene, borneol, and *n*-pentadecane. E. H. Sharples.

Farnesol in jasmine flower oil. F. Elze (Chem.-Ztg., 1926, **50**, 782).—From the residual liquid after separation of the main constituents of jasmine oil a fraction having b.p. 145—155°/5 mm. was isolated. This yielded, on saponification and steam distillation, an alcohol which could be oxidised to an aldehyde giving a semicarbazone of m.p. 134°. No change of m.p. was produced when this substance was mixed with the same derivative of farnesol. Hence the presence of the latter in jasmine flower oil is indicated. A. R. Powell.

Determination of the alkaloid content of lupin seeds by various methods. C. Brahm and G. Andresen (Z. angew. Chem., 1926, **39**, 1348—1350).—The alkaloid content of many samples of lupin meal derived from different varieties of lupins have been determined by the following methods: (*a*) the official German method (cf. Thoms and Michaelis, Jahresber. Ver. angew. Bot., 1918, **16**, 38); (*b*) Sabalitschka and Zaher's method (B., 1924, 653); and (*c*) Mach and Lederle's method (B., 1921, 825 a). Method (*a*) gives very low results when the ether–chloroform extract is washed with water; when this washing is omitted more concordant results are obtained, which are usually lower than those obtained by method (*b*) on seeds from red and white lupins, and higher on seeds from yellow lupins. The results of all three methods, whilst fairly concordant between themselves, differ considerably from one another, and, in the case of method (*c*), the results obtained by the use of the two factors prescribed are different from those calculated from the loss in weight on ignition of the silicotungstate precipitate. A. R. Powell.

Patents.

Manufacture of lactic acid esters. Chem. Fabr. auf Actien (vorm. E. Schering) (E.P. 256,587, 7.7.26. Conv., 7.8.25).—By treating a lactic acid salt with an alcohol in presence of an acid, at a temperature considerably below the b.p. of the alcohol employed, even at room temperature or a little over, good yields of lactic esters are obtained. The addition of an excess of sulphuric acid or of a dehydrating salt, *e.g.*, calcium chloride or magnesium sulphate, is preferred. F. G. Crosse.

Alkylation of aromatic hydrocarbons. P. C. Rushen. From Naugatuck Chem. Co. (E.P. 259,507

7.6.26).—In carrying out the Friedel–Crafts reaction between an aromatic hydrocarbon and an alkyl halide in presence of a metal halide, the heavy oil which separates from the reaction mixture, and which catalyses the reaction, may be reactivated and used repeatedly as catalyst for the reaction without further use of the metal halide if small quantities of the metal are added from time to time. In particular, ethylbenzene is prepared by treating benzene with ethyl chloride in presence of aluminium chloride, separating the oily catalytic mass from the ethylbenzene, adding the mass to fresh quantities of benzene and ethyl chloride, repeating the separation of the oily mass, and so on, maintaining the activity of the mass by occasional additions of aluminium powder or filings so as to form fresh aluminium chloride in the reaction mixture. A. DAVIDSON.

Exothermic catalytic reactions. [Oxidation of naphthalene.] H. D. GIBBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,599,228, 7.9.26. Appl., 27.7.20). —An apparatus for the oxidation of naphthalene to phthalic anhydride is described consisting of a horizontal cylinder containing a number of small catalyst tubes, parallel to the axis, and arranged symmetrically about it. These tubes are packed in a substance with a high thermal conductivity, such as a mixture of sodium and potassium nitrates; at one end they pass into a condenser, and at the other into a manifold. Initially, the apparatus is heated to about 500°, and a mixture of naphthalene and air is introduced into a tube in the axis of the cylinder, in which it is preheated. Thence it passes into the manifold, which has the form of a paraboloid to ensure even distribution, and thence into the catalyst tubes, which contain vanadium oxide, and so to the condenser. All local overheating of the catalyst is avoided. T. S. WHEELER.

Rapid fixing of ethylene by sulphuric acid to obtain neutral ethyl sulphate. A. A. L. J. DAMIENS, M. C. J. E. DE LOISY, and O. J. G. PIETTE (U.S.P. 1,599,119, 7.9.26. Appl., 5.9.23. Cf. B., 1922, 957).—A compound of univalent copper is added, as catalyst, to 97% sulphuric acid, which is maintained at 0—15° while gas containing ethylene is passed through. The catalyst is removed and the acid diluted, when the ethyl sulphate floats on the surface and is collected. A small proportion of ethylsulphuric acid is retained by the sulphuric acid. A. DAVIDSON.

Process for obtaining liquids in a solid form. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 259,431, 29.12.25).—The liquid is mixed with a magnesium alkoxide in the presence of a small quantity of water. The magnesium alkoxide is prepared by adding magnesium to the absolute alcohol and starting the reaction with a trace of bromine or a little ready-made magnesium alkoxide, the temperature being prevented from rising much above the ordinary. For example, 120 pts. of methyl alcohol containing 0·5—1% of water are mixed with a solution of 1 pt. of magnesium in 30 pts. of methyl alcohol. The mixture sets at room temperature in a short time to a solid jelly, which does not melt on heating. Other examples describe the preparation of solid jellies from castor oil and hydrogen cyanide. A. DAVIDSON.

Chlorination of organic compounds [by sulphuryl chloride in presence of catalysts]. O. SILBERRAD, and A. BOAKE, ROBERTS & Co., LTD. (E.P. 259,329, 23.7.25).—The chlorides of phosphorus, manganese, arsenic, selenium, tellurium, antimony, bismuth, molybdenum, bromine, iron, sulphur, and aluminium are used as catalysts, the use of the last two as in E.P. 193,200 (B., 1923, 376) being excluded. Aluminium chloride is used only with aromatic compounds capable of undergoing the Friedel–Crafts reaction. Of the above catalysts, the chlorides of phosphorus, arsenic, manganese, and bromine favour substitution in the side chain in aromatic compounds. For example, 1 lb. of phosphorus pentachloride is dissolved in 106 lb. of *m*-xylene at 70°, 140 lb. of sulphuryl chloride are then added, and the mixture boiled under reflux for 10—12 hrs. Any unchanged xylene and sulphuryl chloride are distilled off, the residue being almost entirely *m*-xylyl chloride. Ring substitution is favoured by the chlorides of antimony, bismuth, iron, molybdenum, aluminium, tellurium and selenium. For example, 1 lb. of antimony trichloride and 92 lb. of toluene are heated to about 70°, and 140 lb. of sulphuryl chloride gradually added. Chlorination is immediate, substitution being entirely in the ring and the yield theoretical. If, instead of using 140 lb. of sulphuryl chloride, 700 lb. are used, pentachlorotoluene is obtained. Sulphur chloride accelerates both types of substitution. The chlorination of acetic acid to monochloroacetic acid in presence of tellurium chloride is described. A. DAVIDSON.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Reversing action of red and infra-red rays on sensitised and fogged photographic plates and the absorption of the sensitising dyestuffs. M. MIYANISHI (Mem. Coll. Sci. Kyoto., 1926, **A10**, 25—29).—On the lines of the investigation of Terenin (A., 1924, ii, 363) the relation between the spectral region reversed by red and infra-red rays and the position of the absorption band of the sensitising dyestuff was studied. Ellington Studio plate was bathed in sensitiser, dried, fogged by candle-light, and exposed in a spectrograph to the spectrum of a carbon arc charged with barium and potassium salts and screened with ruby glass. The spectrum was reversed in the region from λ7500 to λ9000 on the plate treated with Iodo Green, Malachite Green, Methyl Green, Ethyl Violet, Auramine, Nigrosine, and Fuchsine, and in the region λ8500 on the plate treated with Auramine. With Cyanine and Erythrosine no reversal was obtained. The dyestuffs effective in reversing the spectral lines in the infra-red have an absorption maximum in the neighbourhood of λ5500 to λ6000. With Iodo Green, fogging by green light gave best results, recording the lines up to λ9500. With this dye and fogging by violet light, an additional isolated reversal was observed in the region λ6400. W. CLARK.

Sensitivity of photographic emulsions. I. Effect of changing hydrogen-ion concentration during the washing and digestion stages. S. O. RAWLING and J. W. GLASSETT (Phot. J., 1926, **66**, 495—504).—With different samples of gelatin having widely different photographic properties, and with emulsions precipitated in presence or absence of ammonia, the photographic

speed obtained, after digestion, is higher or lower as the emulsions are digested at a higher or lower hydrogen-ion concentration. Fog is, in general, independent of the p_H of digestion. These results held both for pure bromide and iodobromide emulsions. The p_H range studied was from 5 to 9. With any one emulsion the speed on prolonged digestion reaches a steady value which is maintained for a time, and is different for each p_H value employed. The change in speed is thus not merely a question of the rate at which the sensitivity changes during digestion. The size and appearance of grain seem to be unaltered by changes in the p_H of digestion. In most cases, when a sufficiently long digestion period is allowed, the development velocities and shape of the characteristic curve of the emulsions are independent of the changes in hydrogen-ion concentration during digestion. In a given emulsion a change in p_H of digestion alters the sensitivity of each size-class of grains in the same ratio. W. Clark.

Sensitivity of photographic emulsions in relation to quantum energy in exposure. S. E. Sheppard and A. P. H. Trivelli (Phot. J., 1926, **66**, 505—513).—In the case of two emulsions of identical grain-size distribution, one of which was prepared with an "inert" gelatin and the other with a gelatin sensitised with thiourea, the speed values to white light were, respectively, 30 H. and D. and 470 H. and D., while to X-rays the speeds were practically the same. Desensitisation of an ultra-rapid emulsion by chromic acid is slightly greater for red light than for blue light, and almost negligible for X-rays. Below a certain energy level of radiation, sensitisation by "sensitivity centres" is effective, but above this enough energy is conveyed to the silver halide grain within a certain limited area to produce sufficient silver atoms in one nucleus large enough to act as a development centre. The results support the view that the sensitivity centres act as concentration specks for silver in exposure. W. Clark.

Relation between time and intensity in photographic exposure. IV. L. A. Jones and V. C. Hall (J. Opt. Soc. Amer., 1926, **13**, 443—463. Cf. B., 1925, 972; 1926, 566).—With Eastman commercial film and slow lantern plates, gamma infinity and "max. D_∞" (*i.e.*, that value of the density obtained by complete development, which cannot be increased by increase in exposure at the intensity level under consideration) are markedly dependent on the intensity factor of exposure. Eastman par speed portrait film shows the same effects to a lesser degree, as far as measurements have yet been made. Determinations on this film of D_∞ for exposure to constant $I.t$ with various values of I, using light screened with a standard tri-colour red filter, showed the reciprocity law to hold, approximately, in the region of low intensity values, but for values of $I>0{\cdot}14$ mc., the magnitude of density is vitally dependent on the value of I. Materials of the "non-colour-sensitive" type have considerable sensitivity to long wave-lengths when the I factor of exposure is sufficiently great. The values of maximum density obtained in the experiments are shown to represent complete conversion of the silver halide into metallic silver. When the I factor of exposure is below a certain value it is impossible, by extension of time of exposure or development, to make developable all the silver halide grains in an emulsion. This minimum value of intensity, to give complete development of all grains, is roughly inversely proportional to the speed of the material. The silver halide grains must have an intensity threshold, below which it is impossible to produce developability. No emulsions contain "inert" grains provided the intensity factor of exposure is sufficiently high. Smaller grains in an emulsion can reasonably be assumed to have a higher threshold intensity than the larger grains. The effective size-frequency distribution function of an emulsion would then be dependent on the intensity level of exposure, and would differ from the actual size-frequency distribution based on a consideration of all grains present. W. Clark.

Mechanism of the action of light on silver halides. J. Eggert (Z. Elektrochem., 1926, **32**, 491—501).—A review of some recent work.

Patents.

[Photographic] reversal process. J. G. Capstaff, Assr. to Eastman Kodak Co. (U.S.P. 1,600,797, 21.9.26. Appl., 31.1.25).—See E.P. 258,814; B., 1926, 997.

Production of photographic gelatin reliefs. S. de Procoudine-Gorsky and N. Pozniakow (E.P. 246,519, 25.1.26. Conv., 26.1.25).—See F.P. 589,422; B., 1926, 220.

Light-sensitive photographic materials. R. F. Punnett, Assr. to Eastman Kodak Co. (U.S.P. 1,600,736, 21.9.26. Appl., 6.6.24).—See E.P. 235,209; B., 1925, 694.

XXIII.—SANITATION; WATER PURIFICATION.

Aeration of water under open-air conditions. W. E. Adeney (Phil. Mag., 1926, [vii], **2**, 1140—1148).—See B., 1926, 934.

Patents.

Sludge tank and gas producer. H. L. Thackwell (U.S.P. 1,599,731, 14.9.26., Appl., 22.6.25).—A septic tank for sewage purification is V-shaped in cross-section, and contains two longitudinal partitions, which form a hood by extending from abutments at the middle line of the outwardly inclining side walls, upwards and inwards, to meet on the centre line near the level of the top of the tank. Sewage is introduced at one end of the tank, and, after a suitably baffled course, the effluent is withdrawn over adjustable weirs at the opposite end. The gas produced rises into the hood formed by the partitions, which direct it through a screen into a gas chamber, whence it passes to burners, and serves to heat water, which is circulated through pipes in the tank. The heat thus supplied promotes bacterial activity. Means are also provided for removing residual sludge from the bottom of the tank. T. S. Wheeler.

Insecticide. J. S. Cohen and A. B. Leerburger, Assrs. to Interstate Chem. Co. (U.S.P. 1,599,809, 14.9.26. Appl., 15.11.24).—Ethyl carbonate and its homologues have powerful insecticidal properties even in great dilution, and are employed mixed with lime, soap solution, paraffin wax, etc. T. S. Wheeler.

Cleansing and disinfecting compounds. L. E. Whitlock (E.P. 260,326, 2.7.25).

Apparatus for treating liquids and particularly for softening water. United Water Softeners, Ltd., and J. F. Jones (E.P. 259,385, 15.10.25).